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FY 2001 PROGRAMS AND ACCOMPLISHMENTS

MATERIALS RELIABILITY DIVISION

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Reliability of Modern Materials

The Materials Reliability Division develops and applies advanced measurement and modeling methods for evaluating failure modes and mechanisms and investigates the basic physics and materials science of failure. The research focuses on materials and material-related packaging reliability issues in the microscale and nanoscale structures of microelectronic, photonic, and magnetoelectromechanical devices. Other Division projects address materials issues in reliability of large structures, such as bridges, railroads, nuclear reactors, and pipelines.

National Institute of Standards and Technology
Karen H. Brown,
Acting Director

Technology Administration

U.S. Department of Commerce
Donald L. Evans,
Secretary

MATERIALS SCIENCE AND ENGINEERING LABORATORY

FY 2001 PROGRAMS AND ACCOMPLISHMENTS

MATERIALS RELIABILITY DIVISION

Fred R. Fickett, Chief

Thomas A. Siewert, Deputy

NISTIR 6795

September 2001

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Executive Summary

The Materials Reliability Division's mission is to develop and disseminate measurement methods and standards enhancing the quality and reliability of materials and to provide technical leadership in their introduction to appropriate industries. Our main focus now is to meet the need for measurements related to the ever more stringent materials challenges in the microelectronics market. However, our metrology devices and concepts, and the associated materials science base, continue to cover the range of materials from metals to polymers to ceramics and specimen dimensions ranging from the microscale and nanoscale of electronic packages and their components to the massive structures found in gas pipelines and bridges. Many measurement techniques are brought to bear on the problems, ranging from traditional and advanced ultrasonic testing to advanced transmission electron microscopy, scanned probe microscopy, and new measurements yet to be named. The Division also provides measurements and standards to support the instruments necessary for assuring the accurate determination of impact resistance of structural steels through the standard reference materials (SRM) program. In fiscal year 2001 (FY01), the Division pursued the following research areas:

Microscale Measurements: Measurement techniques are developed for evaluating the mechanical, thermal, electrical and magnetic behaviors of thin films and coatings at size scales typical of modern electronic chip and package structures. With our research partners, we continue work to clarify the mechanisms of both on- and off-chip interconnect failure. Robert Keller spent most of the year working at the Max-Planck-Institute for Metals Research on the problem of failure of interconnect lines under ac excitation. Our new NIST test facility, which offers both variable frequency and variable temperature, will be a key part of this continuing project. Scanned probe microscopy continues development as a measurement technique offering the promise of moving to finer scales in acoustic, thermal, and mechanical properties determination. A new instrument with enhanced thermal measurement capability will be in place by year-end, which will extend our capability to smaller scales and greatly help our growing project on evaluation of thermomechanical failure modes of embedded passive devices.

Microstructure Sensing: The use of ultrasonic waves and vibrations to measure the elastic properties of solids continued to be the primary tool for nondestructive characterization of the microstructures that give materials their commercial value. At the high frequency end of the scale, laser excitation and detection of surface acoustic waves allowed the Young's modulus of thin films with thickness values of less than a micrometer to be made with high accuracy. New computational tools to relate the measured ultrasonic wave velocities to the elastic constants of these layered anisotropic materials have to be developed in order to achieve the desired level of accuracy. Surface acoustic waves and ultrasonic vibrations are being used in the atomic force microscope to map, on a submicron scale, the location and extent of the metal and dielectric layers that comprise the micorstructure of modern integrated circuits.

Process Sensing and Modeling: The projects in this area develop measurement technology for determining a material's characteristics and/or implementing real-time process control. In FY01, we continued expansion of our activities into new directions. Our material property database for lead-free solders was significantly expanded and published to our web site. Application of our high-energy x-ray diffraction system to detection and analysis of brittle intermetallic phases in solder joints continued, with apparatus development and testing of the new system. In welding, development of computational models of gas metal arc welding continues. Models of the wire and liner package were developed and verified with experiments. New activities in the Charpy SRM program have resulted in improved customer service to the impact testing community.

Division Chief's Commentary

The change of focus of the Materials Reliability Division to concentrating efforts in the area of electronic materials research continued in FY01, with some success as indicated by the reports contained in this document. We also are making an effort to maintain a presence in the infrastructure support work for which the Division was well known for many years. Staff members continue to be successful in applying their expertise to new types of materials and problems on a significantly different size scale, especially in the areas of thin film elastic properties and thermal transport within electronic packages. We have taken on a few projects that have allowed us to consider new directions for our measurement expertise, such as applications in tissue engineering and nanotechnology. In all areas, we have continued to acquire new apparatus that will allow us to maintain our leadership in development of test techniques for determining material behavior on ever finer scales. Division staff morale and productivity continue at a high level.

Fred Fickett

Technical Highlights

The following Technical Highlights section includes expanded descriptions of research projects that have broad applicability and impact. These projects generally continue for several years. The results are the product of the efforts of several individuals. The Technical Highlights include:

- Alternating Current-induced Cyclic Deformation in Aluminum Interconnects
- Advances in Mechanical Characterization of Thin Films
- Thermomechanical Failure Analysis and Prediction in Microelectronic Structures
- Measuring the Magnetostriction of Thin Films with Ultrasonics
- Ultrasonic Techniques for Measuring the Mechanical Properties of Thin Film
- Determination of Elastic Strain and Stress in Textured Polycrystals by Rietveld Refinement
- Radio Tower Support Failure: Investigation on NIST WWVB Pin

Alternating Current-Induced Cyclic Deformation in Aluminum Interconnects

Reasonable models exist for predicting lifetimes of narrow interconnects during dc stressing, but questions remain as to their applicability to time-varying stressing, which represents an important operating condition. Understanding the mechanisms behind reliability degradation due to alternating currents represents a critical step towards predicting lifetimes under all types of in-use conditions. In this ongoing program, techniques are developed for both testing and characterizing the time-varying current behavior of narrow interconnects. We have found that high current density stressing under ac conditions can lead to thermal fatigue in interconnects, due to differential thermal expansion of the surrounding materials.

Background

The metallic "wires" that connect the elements of electronic devices, carrying the signals and control currents, are called interconnects. They are ubiquitous in modern electronics, with present-day devices containing several kilometers of interconnects. Failure of just one such segment can cause an entire device to function improperly. Interconnects are almost always thin film structures that are prepared by any of a number of techniques depending on their location and function. The larger interconnects (often up to tens of micrometers in width) connect chips and other components on the circuit board. Failure in such lines is usually caused by flexing of the circuit board, thermal stresses, or excess current. At the chip level, the lines are much smaller (presently just a few hundred nanometers wide) and comprise up to seven levels of the structure. They are made of either pure or alloyed aluminum or copper. Chip-level interconnects are the main focus of this program. Failure of these lines tends to occur when voids form either during processing (stress voiding), or in service when carrying currents that may approach a density of one million amps per square centimeter (electromigration). The Division program started several years ago with an emphasis on the problem of stress voiding in copper lines. That work is now considered groundbreaking, evidenced by repeated reference to the labor-intensive results by industrial researchers at conferences addressing chip-level interconnect reliability.

We have recently broadened our focus to the study of current-induced failure, looking at the relationships between highly localized microstructure variations in the lines and the formation of voids, hillocks, and other surface damage. In particular, we felt we could make a more rapid contribution by concentrating on time-varying current stressing, rather than on the already heavily researched field of dc electromigration. The distinction is important, since the vast majority of in-use conditions that microelectronic devices undergo involves both pulsed and alternating currents. Our knowledge of monotonic versus cyclic effects in bulk metals suggests that the corresponding failure mechanisms associated with dc and ac current stressing are also different.

Approach

It was clear at the beginning of the program that understanding interconnect reliability required two experimental activities. The first was the controlled production and identification of damaged regions of interconnects, and the second was measurement and analysis of the crystal and grain boundary structures of such regions and comparison to areas that remained intact. For instance, it has long been known that the main regions of void formation are the junctions of three grains or of two grains and a surface. However, why only certain junctions resulted in voiding was unclear until this effort. At this point, the significance of *local* variations in microstructure became apparent. This realization carries over into present efforts.

We developed in FY00 an electromigration test system in our own laboratory that is more versatile than a commercial apparatus. This allowed us to begin to systematically determine the effects of various electrical and thermal parameters on the failure modes, including both ac and dc testing of lines down to sizes well below one micrometer with variable current, frequency, waveform, and thermal environment. Preliminary ac tests were encouraging, and were followed up by detailed investigations during Bob Keller's nine-month visit to the Max-Planck-Institute (MPI) for Metals Research.

Our experience with cyclic deformation in bulk metals suggested that the specific degradation phenomena controlling ac failure versus dc failure were considerably different. We approached the problem of time-varying current stressing by keeping in mind the concepts of damage accumulation during fatigue of metals. Additional details of the effort are described in the project section of this report.

Accomplishments

The major accomplishments of the program in FY01 center on the identification of a new failure mechanism for microelectronic interconnects. The work was conducted primarily at MPI during FY01, in collaboration with a world-leading group in electromigration.

Interconnect failure due to fatigue during normal use was proposed as a serious threat to reliability. Devices experience many thermal cycles with temperature amplitudes as large as 100 K, due to both current and power cycling. Differences in the thermal expansion coefficients of the different materials comprising a device can lead to changes in stress in the chip-level interconnects on the order of 150 MPa. A stress of this magnitude is sufficient to cause fatigue damage in bulk nanocrystalline metals.

We measured cyclic lifetimes (times to open circuit) associated with stressing at high current density in pure aluminum and aluminum alloy lines of width 0.5 to 13 micrometers. The results are shown in Figure 1.

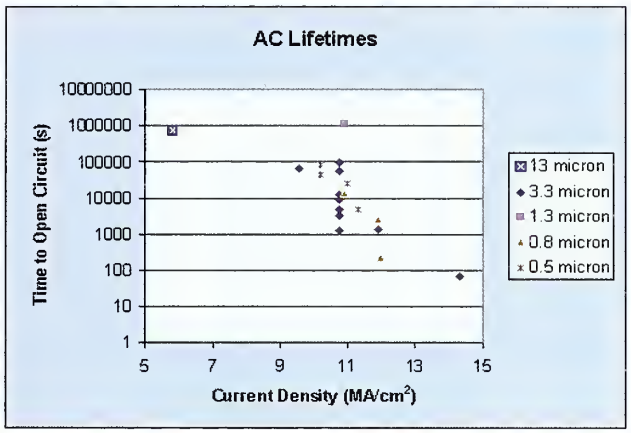


Figure 1. Cyclic lifetime as a function of current density for aluminum and aluminum alloy lines of varying width.

While most of the effort has focused on a current density of approximately 11 MA/cm^2, we see a clear trend in decreasing lifetime with increasing current density. This is unsurprising, but its strong correlation to fatigue descriptions was not expected. At low frequencies, the self-heating of the lines spreads over the entire chip and creates temperature changes of up to 100 K within each ac cycle. This in turn results in thermal strains in the lines. Since current density (squared) tracks with power, and thus with temperature, we can determine a thermal stress associated with each current density. Upon replotting the data of figure 1 into a conventional fatigue stress-lifetime curve, we find that the open circuit behavior follows published fatigue data for nanocrystalline aluminum very closely.

The results suggest that fatigue is indeed the dominant reliability-limiting mechanism during low-frequency ac

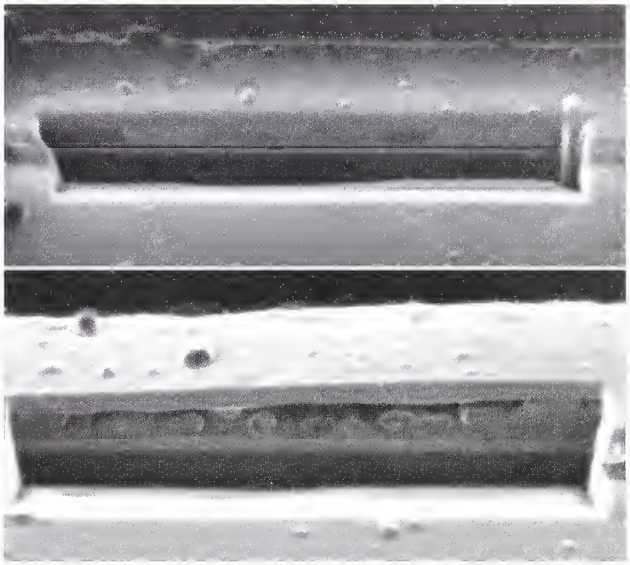


Figure 2. Cross-sectional images of 3 µm wide nitride-encapsulated Al-1Si line before and after ac cycling.

stressing. Open circuit occurs after cyclic deformation has significantly reduced the line cross section. This causes a locally extremely high current density, which then melts the line, leading to failure. We postulate that this has not been seen as a reliability problem to date since interconnects have been mechanically constrained by a hard, strongly adherent encapsulating material such as oxide. Figure 2 shows the effect of encapsulating with a poorly adherent hard nitride encapsulant. Here the damage was not suppressed, and the overlayer bowed and fractured in some places. With the ongoing transitions to very soft, polymeric dielectrics, we expect that fatigue damage in interconnects may not be so easily suppressed. A preliminary test using a soft encapsulant showed that damage was just as severe as that induced in unpassivated lines.

Summary

The results from this new effort have been encouraging. The interest shown by industry and by the MPI indicates that we are very much on the right track to developing an expanding program attacking the issues involved in understanding of the mechanisms of interconnect failure, as opposed to contributing to conventional reliability engineering, which is common in industrial design. The measurement techniques, analysis methods, and data developed here will not only be of immediate value, but will provide solutions to many future problems.

For More Information On This Topic: Robert Keller, Fred Fickett (NIST)

Advances in Mechanical Characterization of Thin Films

With the increasing use of microfabrication techniques to produce commercially promising and important devices such as advanced electronic interconnect and packaging structures, MEMS and NEMS structures, and apparatus for controlled manipulation of biological materials, the mechanical characterization of thin films is becoming increasingly important. The mechanical behavior of the thin films continues to show unexpected differences, both among themselves and compared to bulk behavior. Specimen designs and test methods used in various laboratories for mechanical characterization of thin films appear to be converging.

Background

Advanced electronic devices have for many years derived their impressive performance in extremely small size factors through the use of thin film dielectric and interconnect layers on integrated circuit chips. Recently, electromechanical devices such as moveable mirrors have been manufactured using a related set of thin film technologies. Accurate values for the mechanical properties of these thin film materials are needed by manufacturers for use in mathematical models of their products. These models are used to develop new device capabilities, in electromechanical devices such as moveable mirror arrays, Figure 1, and to assure the reliability of existing devices with minimal use of expensive and time-consuming reliability tests involving large numbers of actual devices. As ever more exotic materials, such as porous low-k dielectrics, are introduced into ever more mechanically demanding designs, mechanical characterization becomes more important. Practically useful mechanical property values cannot be derived theoretically, even for bulk materials, because the properties are so sensitive to the details of the microstructure. It is easier to measure the mechanical properties than to characterize the microstructure in sufficient detail to predict the mechanical properties. Thin films in microelectronic materials, produced by special processes such as physical vapor deposition, have different microstructures from bulk materials with similar chemical composition, and so for them, empirical characterization is the only way to obtain the necessary property values.

Approach

The standard set of mechanical test techniques used for familiar materials with sizes on the order of millimeters is not applicable to microscale materials with sizes on the order of fractions of a micrometer. A special set of test techniques is being developed, at NIST and elsewhere, for characterization of microscale materials. Up until quite recently, the apparatus was designed purely for research, and specimens were designed specifically for a unique set of test apparatus. The specimens were almost always limited to "home-made", because specimen design or necessary fabrication procedures were incompatible with volume manufacturing.

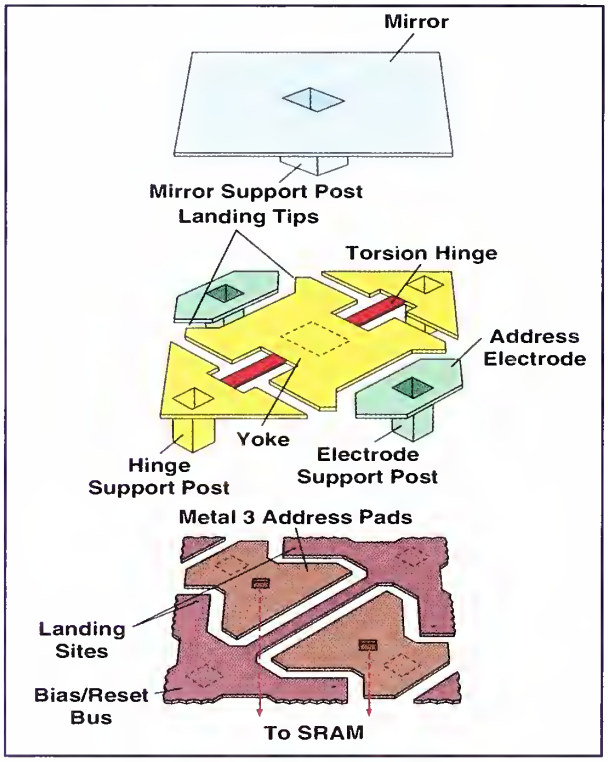


Figure 1. Single pixel in the programmable micromirror array produced by Texas Instruments (schematic diagram).

A fundamental practical issue in wide adoption of a standard mechanical characterization technique for thin films is the use of special test structures or special test chips. The electronics industry prefers to minimize the number of test structures. A similar dilemma exists in the world of bulk materials, where the extra expense of machining standard specimens can be avoided by using hardness tests on raw materials in simple shapes, for example flat plates. However, manufacturers of bulk materials still use tests that require specially made specimens. This is necessary because standard specimen geometries allow unambiguous and accurate determination of mechanical properties, while only conditional correlations exist between a given hardness value and a mechanical property such as ultimate tensile strength.

Accomplishments

The Materials Reliability Division has concentrated on developing specimen designs that are compatible with the microelectronics manufacturing process, and that also allow unambiguous and accurate determination of mechanical properties. We are currently using a specimen with a gauge section 10 µm wide by 200 µm long. Fabrication of this specimen in an arbitrary material has only two requirements: the film must be deposited on bare silicon, and a lithographic patterning procedure must be available. These requirements are easily met in commercial manufacturing processes. This was shown by our recent successful tests of specimens made at a commercial fab through the MOSIS process, Figure 2.

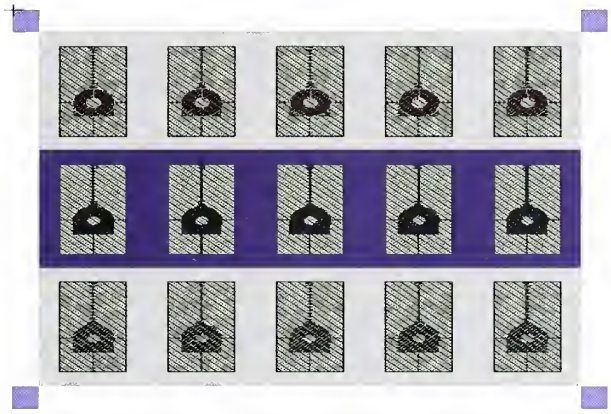


Figure 2. Microfabrication layout of tensile specimens. The gauge section is 10 µm wide, just a fraction of the width of a human hair but much larger than the smallest linewidths currently used in microelectronic devices.

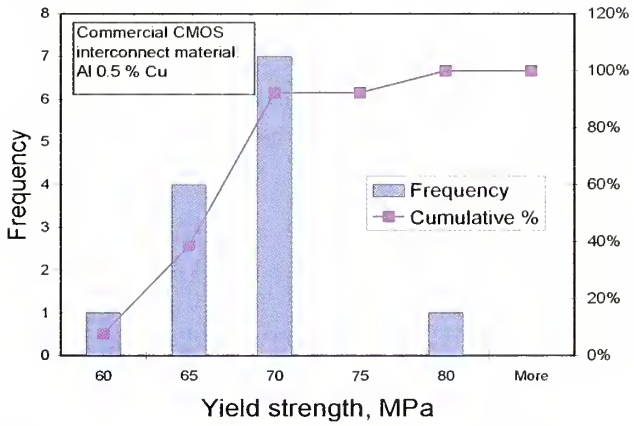


Figure 3. Distribution of yield strength for commercial CMOS material, Al 0.5% Cu.

The specific materials tested were aluminum interconnect layers. The results were surprising in that the strength was very low, Figure 3, and the ductility was also low, as compared to pure aluminum made in our laboratory. Our optimism about the current test technique was bolstered by the discovery that we were able to test the "ring-pull" specimen independently developed and used by Sandia National Laboratories in a recent round robin.

Other components of a robust mechanical testing technique include a practical technique for measuring strains and displacements and the ability to test at temperatures beyond room temperature. We have utilized image correlation in both optical and scanning electron microscopes (SEM) to measure displacements and strains in tensile tests. The technique is practical in that no special specimen treatment is needed, but it still has trouble reaching the small displacements seen in some pathological materials. The technique worked well on pure aluminum and on polysilicon. In the area of testing at elevated temperatures, we are making progress in developing the ability to test at temperature using a heating stage in the SEM.

The current tool set for mechanical characterization of materials makes use of ultrasound for characterizing the elastic behavior of materials at low strains and high strain rates, where plasticity is suppressed. Again, the techniques and apparatus appropriate for bulk materials are not useful for thin films. Progress is being made as reported elsewhere in this report in development of advanced methods of generation and detection of ultrasound in thin films. We are working on a more practical version of the ultrasonic measurement technique that would be applicable for use in test stations commonly used in industry for films on silicon wafers.

Summary

Mechanical testing of thin films continues to be important, and uncertainty about their actual microstructure and its relationship to their mechanical behavior remain. More elements of the tool set used to characterize the mechanical behavior of bulk materials need to be adapted for use on thin films. But the basic test techniques have progressed to a state where specimens designed for one lab can be tested in others. Such "round robin" efforts will clarify the material behavior and will lead to standardization of mechanical characterization of thin films.

For More Information On This Topic: David T. Read, Yi-Wen Cheng and J.D. McColskey (NIST)

Thermomechanical Failure Analysis and Prediction in Microelectronic Structures

Failure in electronic devices usually occurs because of physical damage to the device or thermomechanical degradation of the complex component structures caused by different thermal expansion properties of the wide variety of materials used in the package. Evaluation of the exact failure modes, critical to their resolution, is a difficult problem that becomes increasingly more so as the size of the components and their connections decreases. In this program, a variety of thermal, mechanical, and electron-optical techniques are used to make detailed analyses of the failure of actual packages and the stress buildup that precedes the failure.

Background

Electronic packages are extremely complex structures and becoming more so as the technology of devices advances at a breakneck pace. The physical size of nearly all electronic devices is decreasing rapidly and, at the same time, their capabilities are increasing dramatically. A classic example of this is the cell phone, which now supports capabilities in email, internet access, and soon even operation of vending machines and GPS location and direction finding. Each new capability requires more electronics that must be packed into the existing package.

Computer systems that now operate at speeds into the GHz range require CPU chips and associated memory that operate closely packed elements at very high current densities. The chip structure may have many layers (currently seven is not unusual), and the problem of distributing power and signals among these layers creates materials problems. The large amount of heat generated creates a difficult environment for the elements of the package, and removal of the heat becomes a primary design consideration. Furthermore, as the device is operated, the heating and cooling cycles create large cyclic stresses on the components because of large differences in thermal expansion of the materials.

The list of materials found in modern electronic packages is staggering, ranging from the silicon of the chips to the metals of the interconnects and on to the dielectrics, both polymeric and ceramic. Additionally, the materials that hold the package together and the circuit board structures, which may contain embedded passive devices and certainly contain integral passives, contribute to the complexity. When wireless capabilities are required, additional problems arise because of the high frequencies involved.

When these materials are combined in actual devices, the additional problem of interactions among them further complicate the issue. Both mechanical and thermal measurements must be designed that allow for analysis of the actual behavior of the package. The problem is further complicated, especially in the case of chip packages, by the fact that traditional measurement techniques are not scalable to the sub-micrometer sizes now seen in many applications. Worse yet, the very real possibility of nanometer-scale structures in the near future leaves even the more advanced measurement techniques wanting.

Approach

To aid the microelectronics industry in analyzing how these material interactions affect the integrity of their packages, tests have been developed at NIST. One such technique is electron-beam moiré. This test technique enables one to measure the displacements due to thermomechanical loading in an actual electronic package. To this purpose, a package is sectioned at the area of interest, a radiation-sensitive resist is spun onto the polished cross section, and gratings are etched into the resist using the directed electron beam from a scanning electron microscope (SEM). Following development of the exposed resist, the specimen is ready for testing. The crossed-line grating interferes with the raster of the electron beam in the SEM, thus generating moiré fringes. A comparison of the fringe field between the preloaded and the thermally-loaded images provides the data needed to calculate normal displacements and, from those, normal strains. Shear strains can also be calculated if data are available from both orientations.

When applied to failure analysis and prediction, moiré images can identify strain concentrations (potential failure sites), and interfacial cracks if associated with Mode I or Mode II crack opening. Extended thermal cycling can show how damage is accumulated in the package, indicating which materials are most vulnerable to fatigue failure.

The resolution for displacement using the electron-beam moiré technique is in the range from 50 to 100 nm, depending upon the pitch of the specimen grating. For most packaging applications this resolution is sufficient. However, as packages become smaller, as integration on the die becomes more complex and material failures become significant, and as special low-expansion packages (chip-on-glass) and their associated materials problems become more prevalent, greater resolution will become necessary. Development has begun on two potential solutions. The first is to use a field-emitting SEM (FE-SEM) to perform the lithography function. The electron beam in a FE-SEM is tighter and it is believed that we will be able to halve the pitch of our best current specimen grating. Ultimately, the limitations of electron-beam lithography are such that it cannot provide the grating pitch that we will be requiring in the future. For that purpose we have turned our attention to nature and

the atomic force microscope (AFM). The outer protein layer of bacteria forms a regular crystalline array (Figure 1). This array can be used directly or as a mask for the specimen grating. The raster of the probe tip of the AFM interferes with this grating to generate moiré fringes. At this point, it is not clear whether a commercially available AFM is able to raster precisely enough to produce quantitative moiré data. Preliminary results would indicate that some modifications to a metrology (closed-loop) AFM system may be necessary to obtain the required precision.

The success of our capability in measurements with thermal microscopy has led to industrial interaction. We are measuring interfaces between new materials used for embedded passive applications. We have added quantitative measurement of interfacial thermal resistance across interfaces and we can calculate interfacial thermal resistance as a function of thermal cycling for analysis of damage iniation and evolution in embedded passive applications. Thermal imaging using scanned-probe microscopy (SPM) has been added, allowing us to view heat flow corresponding to that of the IR microscopy system, but at the increased spatial resolution of the SPM. Development of this capability is continuing with the acquisition of equipment that will allow for measurement of temperature and heat flow using SPM.

Accomplishments

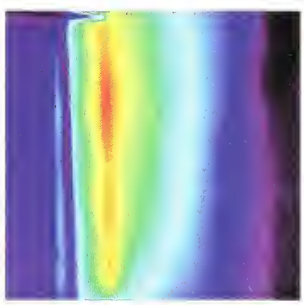
Software was developed at NIST that provides a quantitative measurement of interfacial thermal resistance across interfaces in electronic packaging and embedded passive materials. We analyzed a set of images to track damage evolution as a function of thermal cycling in materials used for embedded resistor applications. We measured one set of integral resistors from an industrial supplier using both IR microscopy and thermal SPM (see Figure 2). We began a collaboration with a manufacturer of embedded resistors and have made measurements on a number of materials used for embedded resistor applications by both IR microscopy and thermal SPM. We are helping U.S. industry by providing unique measurement capabilities consistent with their needs.

Extensive evaluation of AFM systems from different manufacturers was conducted for applicability for scanning thermal microscopy and scanned-probe moiré. A system was chosen, purchased, and delivered to NIST. Electron-beam moiré tests were conducted on column grid array (CGA) specimens from IBM in Rochester, MN, and embedded resistive elements for MicroFab Technologies and SAS Circuits in association with Dupont. The solder columns of the CGA specimen connect a ceramic to printed circuit board (PCB), two materials with vastly differing coefficients of thermal expansion. IBM asked us to determine the shear strains at the interfaces between the solder and each, the ceramic and the PCB. For the embedded resistors, the manufacturers are concerned with the strain experienced by the termination material on the resistors. We were asked to evaluate the strains in this region.

A computer program was written in-house to control the location and dwell time of the electron beam in the FE-SEM to enable the equipment to be used for electron-beam lithography. We are now in the process of determining how the dose needs to be modified and what minimum pitch is attainable.

Summary

The success of the program and the resulting interest from industry has been excellent this year. Our ability to measure the next several size generations of devices in all research areas means that the work will continue to be of value for some time to come. Providing industry with the vital data they need, staying up with their current needs, and ahead of projected needs continue to be our goals.



Left - Figure 1. AFM image of bacteria protein layers showing regular hexagonal array. Right - Figure 2. Scanning thermal-microscopy image of integral resistor from MicroFab Technologies.

For More Information On This Topic: Andrew Slifka, Elizabeth Drexler (NIST)

Measuring the Magnetostriction of Thin Films with Ultrasonics

Thin films of magnetic materials have many applications in microelectronics and nanotechnology. Their magnetostriction plays an obvious role in mechanical actuators for MEMS devices and it can have both deleterious and beneficial effects when used in magnetic memory structures. We are developing an ultrasonic technique for measuring the magnetostriction coefficient of a local region of a magnetic film when it is part of a device or an element of a combinatorial library. If successful, the technique could lead to a device to perform on-wafer mapping of magnetostrictive properties.

Background

Magnetic memory systems demand careful control of the magnetostriction of the thin layers used to achieve the desired performance throughout the system. Furthermore, MEMS technology often uses magnetostrictive films for electromechanical transducers. In order to develop thin films with optimal magnetostriction coefficients for a particular application, the conventional procedure is to measure the deflection of a microscale cantilever beam that has been specially prepared for the experiment. This is both expensive and may not be fully characteristic of the film used in a device. It is also not compatible with combinatorial methods for the rapid screening of many materials for their magnetomechanical properties. If successful, the technique could lead to a device to perform on-wafer mapping of magnetostrictive properties.

Figure 1. Photograph of the apparatus used to compare the theoretical model for a MUT with measurements on a combinatorial library formed by a Ni film on silicon.

The Magnetostrictive Ultrasonic Transducer (MUT)

The Materials Reliability Division has had a long-standing program to investigate various kinds of noncontacting ultrasonic transducers for nondestructive testing applications, as process control sensors and to perform research in materials characterization. When magnetic materials have been involved, magnetostriction provided a transduction mechanism that offered enhanced coupling efficiency and the ability to excite and detect special ultrasonic wave modes. Theoretical models to describe the operation of MUTs on steel were developed in the 1970's and were used in many industrial applications during the 80's and 90's. One prediction of these models was that a surface-skimming shear wave passing under a coil held near the surface could induce an electrical signal in that coil that was proportional to the magnetostrictive coefficient of the material under the coil. Figure 1 shows an apparatus designed to exploit this prediction and enable the rapid collection of data on the magnetostrictive coefficient of a combinatorial library of a magnetic thin film. The figure shows a circular silicon wafer covered with a thin magnetic film that has been divided up into many individual library elements. A flat "meander line" coil connected to a strip-line sits just above one of the library elements. When the conventional ultrasonic shear wave transducer shown on the bottom of Figure 1 is coupled to the left hand edge of the wafer and activated with a short tone-burst of electrical energy, a signal is detected in the coil whose time of arrival is determined by the separation distance between transducer and the coil divided by the velocity of sound in the wafer. The signal's amplitude is proportional to the electromagnetic skin depth in the film multiplied by the magnetostrictive coefficient of the library element divided by the magnitude of an external magnetic field applied parallel to the plane of the silicon wafer.

Accomplishments - Magnetostriction of Sub-micron Ni Films

Since application of this ultrasonic method to thin films is new, the FY01 program focused on making measurements on a film with known magnetostrictive properties. The metal chosen was pure nickel and the library was constructed on a silicon wafer with the film thickness as the parameter that varied between the elements of the library. Since the ultrasonic frequency used for these experiments was 5.8 MHz, the electromagnetic skin depth was estimated to be approximately 20 microns which exceeded the thickness of the film. Thus, the skin depth parameter in the theory can be replaced by the film thickness and the theoretically predicted magnetostriction coefficient should be proportional to the ultrasonic wave amplitude multiplied by the magnetic field and divided by

the thickness. Figure 2 shows the amplitude of the signal from the EMAT coil as a function of the applied magnetic field. At high fields, the signal becomes small as expected from the 1/H dependence predicted by the theory. There are zeros in the ultrasonic wave amplitude near zero in the applied field caused by magnetic hysteresis.

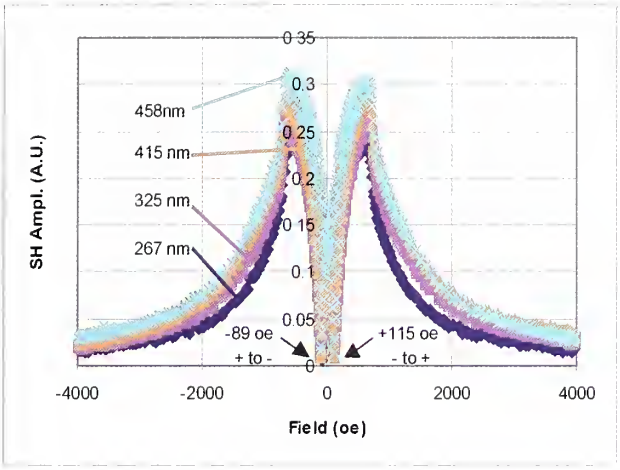


Figure 2. Plot of the signal amplitude vs field for a Ni film on Si. The different thickness values correspond to different library locations.

Figure 3 shows the results of applying the theory to the experimental measurements shown in Figure 2. Here the signal amplitude multiplied by the field divided by the film thickness is shown as a function of applied field. This parameter should mimic the magnetostrictive behavior of pure nickel taken from the literature displayed as a solid line in Figure 3. The figure shows that this expectation is qualitatively correct in that the ultrasonic measurements appear to become field independent at high fields just as is the case for bulk nickel. The approach to saturation appears to be more rapid in bulk nickel than in the films and there is an indication that some library elements have a lower magnetostriction than bulk nickel in the high field limit.

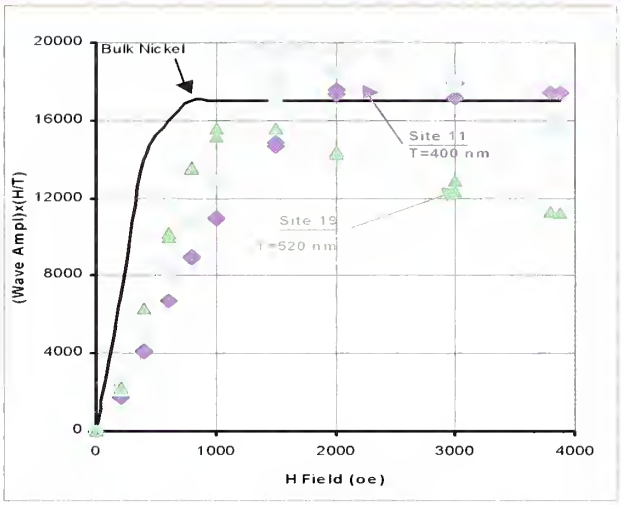


Figure 3. Comparison of the magnetic field dependence of the magnetostriction coefficient of nickel with predictions of the theoretical model applied to a Ni film on silicon.

For More Information On This Topic: G.A. Alers (NIST) and S. Russek (NIST, Div. 814)

Ultrasonic Techniques for Measuring the Mechanical Properties of Thin Films

In order to meet the needs for new materials, it is becoming common practice to use coatings and thin films on special substrates. This is especially true for microelectronic devices where films with desired electric, magnetic or mechanical properties are deposited on single crystal wafers of silicon. Measurements of the elastic constants of these films are important for estimating the residual stress built up in the films during processing as well as during in-service thermal cycling. Furthermore, the microstructure of the film can be monitored and verified by measuring its elastic properties.

Technical Description

During the past several years, a few very accurate ultrasonic techniques for measuring all of the elastic moduli of single crystals and composites have been developed in order to characterize solids that are elastically anisotropic. The ultrasonic frequencies employed ranged from 100 kHz to 20 MHz and thus covered a wavelength range from centimeters down to millimeters. Thus, the samples used were usually in bulk form and had a similar range of dimensions. We have now embarked on a program to extend the ultrasonic methods to materials of interest in the microelectronics industry. These materials are in the form of thin films with thickness dimensions extending down to well below a micron (10^{-3} mm) and whose commercial value stems from their highly anisotropic physical properties. Although some laboratories are approaching this problem by simply extending conventional pulse-echo techniques into the femtosecond timing range, our approach is to exploit our well-developed expertise in ultrasonic wave propagation through materials whose microstructures are on a scale small compared with the acoustic wavelength. In this way, we can utilize well established ultrasonic methods that are powerful because they employ a wide variety of wave types and propagation directions to develop many independent relationships between different unknown parameters and several measurable quantities.

In addition to the focus on thin films used in the microelectronics industry, the models and measurement techniques developed in this program can also be used to characterize surface coatings and layers with thickness values greater than 100 microns. For example, thermal barrier coatings, wear-resistant layers, shot-peened surfaces and case-hardened shafts are commonplace in heavy industry but are difficult to characterize because only x-rays of very high energy or neutrons can interrogate the full depth of these "thick" layers. Another industrially important surface property is the residual stress left in rolled plates and forged products by their manufacturing processes. When these materials are machined into precision parts, the residual stresses are relieved and the dimensional tolerances of the part can be compromised. Ultrasonic methods of measuring surface residual stress gradients are being developed for use in rolling mills or forging shops to insure the adequacy of stress relief anneals before expensive parts are machined.

Approach

Two different approaches are being followed to measure the elastic properties of thin films. One approach uses surface acoustic waves (SAWs) that propagate parallel to the surface partly in the film and partly in the substrate. The other uses Atomic Force Microscopy (AFM) techniques modified by the addition of ultrasonic vibrations. Both these approaches require the development of new mathematical models to relate the measured quantities to the elastic properties of the layer.

Accomplishments – Models

Models for analyzing the propagation of SAWs on layered media have been well developed for the design of commercial devices used as electronic resonators and filters (i.e., for SAW devices). Because they are used for device design, these models are well suited for the forward problem of predicting the velocity of a surface wave from inserted values of the dimensions and elastic properties of the film and substrate. The models being developed for our program put the emphasis on the inverse problem of deducing the dimensions and elastic properties of the film from measurements of the surface wave velocity and an accurate knowledge of the substrate properties. We have applied a dynamic Green's function formalism to the development of a rapid and computationally efficient way to predict the velocity of a surface wave propagating on an anisotropic substrate supporting an anisotropic film whose thickness is much less than the wavelength. Because of the computational efficiency of the model, a rapid inversion procedure is now available for deducing a few of the film properties from measurements of the frequency dependence of the velocity of the surface wave as a function of crystallographic direction in the substrate. During FY 01, extension of the model to low frequencies where the surface wave becomes a Lamb wave in the substrate has exposed several interesting features that can be used to deduce additional properties of the film.

Accomplishments – SAW Measurements

During FY01, the pulsed laser ultrasonic wave generator and the Michelson interferometer system for excitation and detection of surface waves on single crystal plates were perfected and calibrated by studying pure aluminum and molybdenum films on fused silica substrates. Measurements of the velocity of surface waves

propagating on silicon substrates covered with TiN films in the thickness range of 100 to 500 nm were carried out at ultrasonic frequencies in the range of 20 to 400 MHz. Analysis of these data demonstrated the power of the Green's function model by producing values for two elastic constants of the anisotropic films as well as their thickness values which were subsequently verified by destructive tests using an SEM. This program is being carried out in cooperation with CSIRO in Australia who prepared the samples under a variety of deposition conditions designed to put different residual stresses into the films. As a further check on our methods, we are working to compare our results with those using nanoindentation methods at NIST-Gaithersburg as well as at BAM (Berlin).

Accomplishments – AFAM Measurements

The Atomic Force Acoustic Microscope (AFAM) assembled last year was designed to measure the shift in resonant frequency of the cantilever as it approached the surface of a sample. A model to relate this frequency shift to the Young's modulus of the surface is being developed and was tested on an aluminum film 1 mm thick. With the AFAM, we obtained a value of 67 ± 7 GPa for the Young's modulus of the film. This compares favorably with literature values of 67 to 71 GPa for bulk aluminum. A value of 68.6 ± 0.2 GPa was obtained on the same film using surface acoustic wave methods. By holding the excitation frequency constant and measuring the cantilever's vibration amplitude as the tip was scanned across the sample, 2-D maps of the surface modulus have been prepared. We have obtained preliminary elasticity images of a damascene copper/SiO_2 dielectric test structure used by the microelectronics industry. Here, the area covered was 2 µm x 2 µm and the 100 nm wide stripes were clearly resolved.

Accomplishments – Thick Layers

In many important industrial applications, the surface film or layer with the desired properties may have a thickness that exceeds 100 µm and have a gradient in properties through that thickness. During FY01, a program to apply the techniques developed for measuring very thin film properties to the characterization of such surface layers on structural materials was initiated. It was motivated by the industrial need for nondestructive testing methods to monitor the quality of surface treatments designed to extend the life of machinery or minimize the distortion of precision parts during final machining. By generalizing the Green's function formalism to describe sub-surface gradients in physical properties as a stack of individual thin films and by using electromagnetic transducers (EMATs) that are capable of generating and detecting several different types of surface-skimming acoustic wave types, we anticipate that a residual stress gradient can be detected and measured even if there is a gradient in preferred orientations (i.e. a texture) in the grains near the surface of the material. To test this contention, very accurate measurements of the phase velocity of surface-skimming shear horizontal (SH), longitudinal (SSL)and Rayleigh (SAW) waves are being collected as a function of applied stress and propagation direction on commercial aluminum alloy plates. These are expected to have different rolling textures and stress gradients as a function of depth below the surface. The figures below summarize some of these results.

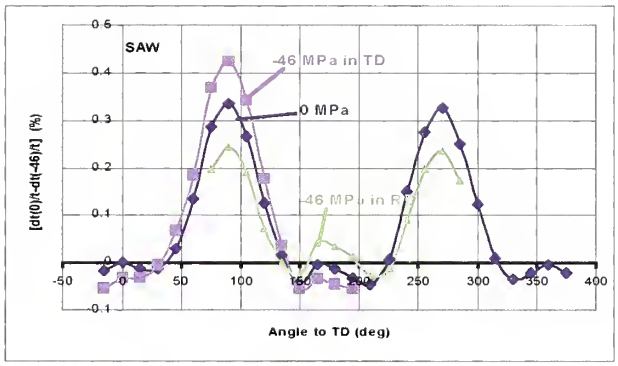


Figure 1. Fractional shift in transit time of a surface wave propagating over a fixed distance at various angles to the transverse direction (TD) and for different loads.

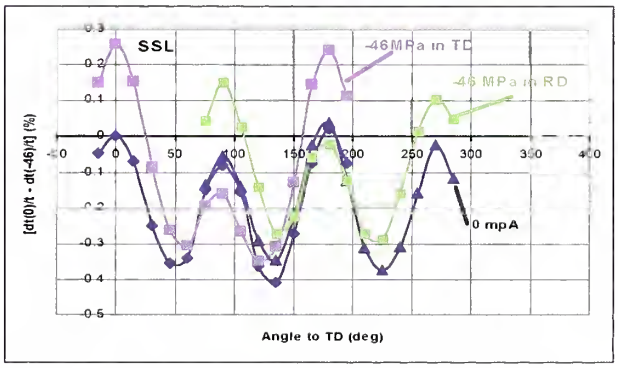


Figure 2. Fractional shift in transit time of a surface-skimming longitudinal (SSL) wave vs direction relative to the transverse direction (TD) for different loads.

For More Information On This Topic:

V. Tewary, D. Hurley, G. Alers (NIST)
A. Richards (CSIRO)
R.B. Thompson (QNDE Center, ISU)

Determination of the Elastic Strain and Stress in Textured Polycrystals by Rietveld Refinement

We developed a novel approach to model diffraction line shifts caused by elastic residual or applied stresses in textured polycrystals. The model yields the complete strain and stress tensors as a function of crystallite's orientations, as well as the average values of macroscopic strain and stress tensors for arbitrary crystal symmetry.

The stress state (where the stress can be both applied and residual, that is, resident in the material after an external force is removed) influences many different materials properties, some of which are especially important in diverse engineering and technological applications. For instance, stress has a large influence on durability and longevity of bridges, buildings, and other structural components. Moreover, stress influences the operation of modern electronics, such as satellite and wireless communication devices, and computers. X-ray and neutron diffraction are the most accurate and widely used methods for stress determination in crystalline materials.

A common approach for strain and stress determination uses the method where the strain is derived from directional measurements of the crystal interplanar spacing, ***d***, as a function of the angle between the diffraction vector and an arbitrary direction in the specimen. Recently, it was proposed that the strain/stress orientation distribution function (SODF), which is defined as a strain/stress tensor component as a function of a crystallite's orientation, be determined through the expansion in series of generalized spherical harmonics. Instead, we based our approach on a development of the texture-weighted strain orientation distribution function (WSODF) ε_i' in a series of generalized spherical harmonics P_l^{mn}:

$$\varepsilon_i'(\varphi_1,\Phi_0,\varphi_2)=\sum_{l=0}^{\infty}\sum_{m=-l}^{l}\sum_{n=-l}^{l}c_{il}^{mn}\exp(im\varphi_2)P_l^{mn}(\Phi_0)\exp(in\varphi_1)$$

Here $(\varphi_1,\Phi_0,\varphi_2)$ are the Euler angles transforming the sample orthogonal coordinate system into the crystallite's orthogonal coordinate system. The reason to directly determine WSODF instead of SODF is in accord with the fact that diffraction experiment yields the texture-weighted strain, which is also used to calculate average strain $\boldsymbol{e}_i$ and stress s_i tensors in the sample coordinate system

$$\bar{e}_i=(1/8\pi^2)\int_0^{2\pi}\int_0^{\pi}\int_0^{2\pi}d\varphi_1 d\Phi_0 d\varphi_2\sin\Phi_0 f(\varphi_1,\Phi_0,\varphi_2)e_i(\varphi_1,\Phi_0,\varphi_2)$$

$$s_i=(1/8\pi^2)\int_0^{2\pi}\int_0^{\pi}\int_0^{2\pi}d\varphi_1 d\Phi_0 d\varphi_2\sin\Phi_0 f(\varphi_1,\Phi_0,\varphi_2)s_i(\varphi_1,\Phi_0,\varphi_2).$$

Averaging is weighted by the crystallite orientation distribution function (CODF) $f(\varphi_1,\Phi_0,\varphi_2)$. The following linear relations link the strain and stress tensor components in the sample and crystallite coordinate systems:

$$s_i=\sum_{j=1}^{6}P_{ij}\sigma_j\quad e_i=\sum_{j=1}^{6}P_{ij}\varepsilon_j\quad \sigma_i=\sum_{i=1}^{6}C_{ij}\rho_j\varepsilon_j$$

The third relationship states the Hooke's law relationship in the crystallite coordinate system.

This approach for determination of strain and stress tensors is particularly suitable for introduction in whole powder-diffraction-pattern modeling approaches, such as Rietveld-refinement programs. An advantage of this approach is that all available Bragg reflections are used simultaneously to obtain the strain tensor. The approach consists of the estimation of the coefficients in the Rietveld program to yield WSODF and the average strain tensor. The average stress tensor can also be determined if the monocrystal elastic stiffness moduli C_{ij} are known. The required number of refined coefficients to achieve the desired precision of WSODF, strain, and stress tensors will depend on the crystal and sample symmetries, as well on the magnitude and gradient of the strain and texture. Even if the strain/stress determination by Rietveld refinement is not of interest, diffraction line shifts caused by residual stresses will generally be crystal-direction dependent. Without correction for this effect, the diffraction line shifts would preclude accurate determination of the structure and refinement using the Rietveld approach, pole figure (texture) measurements, and similar tasks. However, for a successful application in the Rietveld refinement, the challenge lies in the accurate modeling of strain and stress dependence on the crystallographic direction and the ability to handle different crystal symmetries. We extended this formalism to an arbitrary crystal symmetry and gave selection rules for all Laue classes by postulating that the observable quantity measured by diffraction in a given sample direction, that is, the interplanar spacing ***d***, averaged around the diffraction vector, is invariant to the point-group symmetry operations.

Though mathematically similar, there is an important difference from previous approaches in the manner in which crystal and sample symmetries are applied to the coefficients of spherical harmonics. Because of the boundary conditions, two non-spherical crystallites under a given stress could have different strain states, even if they have crystallographically equivalent orientations. In another words, every element of the strain tensor is not necessarily invariant to the crystal symmetry operations, as it was assumed in previous approaches. On the contrary, the observable quantity measured by diffraction in a given sample direction, that is, the interplanar spacing averaged around the diffraction vector, is invariant to the crystal symmetry operation. We used this invariance condition to derive the selection rules in the WSODF harmonic representation for all Laue classes. The selection rules for crystal symmetry for WSODF are different from those for the texture.

For textured samples under stress, two sample symmetries must be distinguished: texture and stress/strain sample symmetry. Sometimes they are identical, but generally the strain sample symmetry can be lower than the texture sample symmetry. Furthermore, the texture sample symmetry operations must form a supergroup of the strain sample symmetry point group. One example is where one considers the uniaxial stress, acting on the sample with a cubic sample texture. The symmetry for strain is tetragonal if the stress axis is along the cube axis, trigonal if the stress axis is along the body diagonal, but triclinic if this axis is oriented in an arbitrary direction. Concerning the selection rules for the harmonics coefficients of WSODF, they are identical to those for the texture of the same sample symmetry.

For the calculation of both average elastic strain and stress tensors, only the harmonic coefficients of WSODF with the indices $l = 0$ and $l = 2$ are necessary. This is a direct consequence of the orthogonality of the spherical harmonics combined with the nature of the transformation matrix for the second rank tensors. Therefore, keeping in expression for strain only the terms with $l = 0$ and $l = 2$ and rearranging to have only positive indices m, n in place of ε_i' we have the following:

$$\varepsilon_i''(\varphi_1, \Phi_0, \varphi_2) = \sum_{k=0}^{25} g_{ik} R_k(\varphi_1, \Phi_0, \varphi_2)$$

The functions $R_k(\varphi_1, \Phi_0, \varphi_2)$ are linear combinations of $\cos(m\varphi_2 \pm n\varphi_1) Q_l^{mn}(\pm\cos\Phi_0)$ or $\sin(m\varphi_2 \pm n\varphi_1) Q_l^{mn}(\pm\cos\Phi_0)$ terms, where

$Q_l^{mn} = P_l^{mn}$ for $m + n$ even and $Q_l^{mn} = iP_l^{mn}$ for $m + n$ odd.

For average strain and stress, one obtains:

$$\bar{e}_i = \sum_{j=1}^{6} \sum_{k=0}^{25} \overline{W}_{ijk} g_{jk}.$$

$$\bar{s}_i = \sum_{j=1}^{6} \sum_{k=0}^{25} \overline{W}_{ijk} g'_{jk}, \text{ where } g'_{jk} = \sum_{l=1}^{6} C_{jl} g_{lk} \text{ and } \overline{\mathbf{W}} \text{ is}$$

$$\overline{W}_{ijk} = (1/8\pi^2) \int_0^{2\pi} \int_0^{\pi} \int_0^{2\pi} d\varphi_1 d\Phi_0 d\varphi_2 \sin\Phi_0 P_{ij}(\varphi_1, \Phi_0, \varphi_2) R_k(\varphi_1, \Phi_0, \varphi_2)$$

In the case when finding WSODF and the average strain and stress tensors during the Rietveld refinement is not of interest, one can choose a different representation with fewer refinable parameters that corrects only for the shifts caused by stress. This alternative representation uses for the dependence of the shift on the direction in crystal, homogenous polynomials in direction cosines, which are invariant to the Laue group operations. Finally, if the average strain and stress are desired but not WSODF, the optimal choice is a hybrid representation for the line shift, with spherical harmonics for $l = 0,2$ and homogenous polynomials for $l \geq 4$.

This novel approach is an improvement of the measurement and analysis capabilities for the determination of residual strain and stress in materials. Moreover, we expect that it will significantly contribute to a successful solution of some industrial problems, where a speedy and accurate determination of strain/stress state in multiphase material components is required. A peer-reviewed full paper was published and work presented at a few meetings. We are currently working on applying this methodology to several problems of interest to industry and research community.

For More Information On This Topic: Tom Siewert, Davor Balzar, Nicolae C. Popa (NIST)

Radio Tower Support Failure Investigation on NIST WWVB Pin

The NIST operated WWVB tower in Ft. Collins, Colorado partially collapsed as a result of an insulator pin failure. The Materials Reliability Division investigated the failure of the pin to determine why the failure occurred after only 2 to 3 years of operational service. The results of this study will be used for the design of new insulator pins to prevent a recurrence of the problem.

The failure of an insulating support pin was responsible for the collapse of the uppermost 30 meters of the 122-meter NIST WWVB transmitter tower in Ft. Collins, CO; see Figure 1. The tower continuously broadcasts time and frequency signals at 60 kHz throughout North America. These signals automatically synchronize electronic products such as wall clocks, clock radios, and wristwatches used by millions of people in the U.S. In addition, the signals are used for high-level applications such as network time synchronization and frequency calibrations. The support pin was one of four groups of nine that support the four towers that make up the WWVB transmitter facility. The tower, originally constructed in 1962, was refurbished in 1999 when the pins, used to accommodate an insulator on the guy wires, were replaced; see Figure 2. Thus, the failed pin had been in service only 2 to 3 years compared to 37 years for the old pins. In order to assess the cause of this premature failure, the Materials Reliability Division conducted a detailed analysis. This Division has been involved in numerous other failure investigations. A rapid assessment was needed, since WWVB is a highly visible NIST service, and a return to operation at full power was necessary to maintain nationwide coverage. We recommended the removal of all the "new" pins and replacing them with the "old" pins until the investigation was completed.

Figure 1. Photo shows results of failure of insulator pin. The uppermost 30 meters of the 122- meter tower has collapsed.

Figure 2. Close-up of insulator and pin arrangement. Pin length is 61 cm.

Initial examination of the failed pin showed that failure was initiated by fatigue. The fatigue crack was located in the radius of the head-to-shank fillet of the pin, Figure 3. The pin is large, 38 mm in diameter with a 51 mm head. Visual, chemical and microscopic metallurgical examinations of the pin were performed. The conclusion was that a fatigue crack initiated at the fillet and propagated across the diameter of the pin body until the remaining area of the pin was no longer capable of sustaining the loads imposed by the tower, with the resulting catastrophic failure. This occurred because the new pin had a fillet radius approximately half that of the old pins, increasing the stress concentration in the shank-to-head transitional region. In addition, circumferential tool marks, tool chatter and surface pitting had further increased the stress concentration that led to the fatigue failure.

Figure 3. The failed pin and pin head showing fatigue fracture surface.

This information will form the basis for specification and production of a new set of pins. In the meantime, the old pins have been reinstalled, a new top section of the tower put in place, and WWVB is back on the air at full power. The information generated will be useful for future evaluations of other installations – there are many radio towers of similar design around the country.

For More Information On This Topic: J.D. McColskey, C.N. McCowan, R.L. Santoyo (NIST)

Materials for Microelectronics

Today's U.S. microelectronics and supporting infrastructure industries are in fierce international competition to design and produce new smaller, lighter, faster, more functional, and more reliable electronics products more quickly and economically than ever before.

Recognizing this trend, in 1994 the NIST Materials Science and Engineering Laboratory (MSEL) began working very closely with the U.S. semiconductor, component and packaging, and assembly industries. These early efforts led to the development of an interdivisional MSEL program committed to addressing industry's most pressing materials measurement and standards issues central to the development and utilization of advanced materials and material processes within new product technologies, as outlined within leading industry roadmaps. The vision that accompanies this program – to be the key resource within the Federal Government for materials metrology development for commercial microelectronics manufacturing – may be realized through the following objectives:

- Develop and deliver standard measurements and data;
- Develop and apply *in situ* measurements on materials and material assemblies having micrometer- and submicrometer-scale dimensions;
- Quantify and document the divergence of material properties from their bulk values as dimensions are reduced and interfaces contribute strongly to properties;
- Develop models of small, complex structures to substitute for or provide guidance for experimental measurement techniques; and
- Develop fundamental understanding of materials needed in future microelectronics.

With these objectives in mind, the program presently consists of twenty separate projects that examine and inform industry on key materials-related issues, such as electrical, thermal, microstructural, and mechanical characteristics of polymer, ceramic, and metal thin films; solders, solderability and solder joint design; photoresists, interfaces, adhesion and structural behavior; electrodeposition, electromigration and stress voiding; and the characterization of next generation interlevel and gate dielectrics. These projects are conducted in concert with partners from industrial consortia, individual companies, academia, and other government agencies. The program is strongly coupled with other microelectronics programs within government and industry, including the National Semiconductor Metrology Program (NSMP) at NIST.

FY2001 Projects (and division leading project)
Lithography/Front End Processing
Characterization of Ultrathin Dielectric Films (Ceramics)
Lithographic Polymers (Polymers)

On-chip Interconnects
Interconnect Materials and Reliability Metrology (Materials Reliability)
Measurements and Modeling of Electrodeposited Interconnects (Metallurgy)
Thin Film Metrology for Low K Dielectrics (Polymers)

Packaging and Assembly
Packaging Reliability (Materials Reliability)
Solder Interconnect Design (Metallurgy)
Solders and Solderability Measurements for Microelectronics (Metallurgy)
Tin Whisker Mechanisms (Metallurgy)
Wafer Level Underfill Experiment and Modeling (Metallurgy)
Wire Bonding to Cu/Low-k Semiconductor Devices (Metallurgy)
X-ray Studies of Electronic Matls. (Materials Reliability)

Crosscutting Measurements
Dielectric Constant and Loss in Thin Films and Composites (Polymers)
Electron Beam Moiré (Materials Reliability)
Ferroelectric Domain Stability Measurements (Ceramics)
Measurement of In-Plane CTE and Modulus of Polymer Thin Films (Polymers)
Mechanical Properties of Thin Films (Ceramics)
Permittivity of Polymer Films in the Microwave Range (Polymers)
Polymer Thin Films and Interfaces (Polymers)
Texture Measurements in Thin Film Electronic Materials (Ceramics)
Thermal Conductivity of Microelectronic Structures (Materials Reliability)

Contact Information: David T. Read (NIST)

Mechanical Behavior of Thin Films

David T. Read

Techniques for characterizing the mechanical behavior of thin films are being developed and applied. Because the films are formed by physical vapor deposition, their microstructures, and hence their mechanical properties, are quite different from those of bulk materials of the same chemical composition. While the general principles of conventional mechanical testing are applicable to thin films, special test equipment and techniques are required. The ultimate goal of this project is to test specimens produced by semiconductor fabrication equipment and similar in size to features on integrated circuit chips.

Technical Description

Thin films are an essential component of all advanced electronic devices. Understanding of failure modes in these devices, especially interface delamination, requires knowledge of the mechanical behavior of the films. Techniques for measuring the mechanical behavior of thin films are being developed and applied.

The objectives of this project are:

- to develop experimental techniques to measure the mechanical properties of thin films, including basic tensile properties, fatigue, and fracture resistance, in specimens fabricated and sized like materials used in actual commercial devices;
- to relate thin film mechanical behavior to microstructure;
- to extend test techniques from their present level (1 µm thick, 10 µm wide) to smaller specimens that are similar in size to the conductive traces used in contemporary VLSI circuits (widths of 0.1 to 1 µm).

Accomplishments

Last year's report introduced the new force-probe tensile test technique. This year we applied the technique to two new types of materials: aluminum interconnect layers made in a commercial CMOS (complementary metal oxide silicon) fabrication facility, obtained through the MOSIS service, and polycrystalline silicon made at Sandia National Laboratories. The key feature of these specimens is their small size; both the pull ring in the polycrystalline silicon specimen, Figure 1, and the hole in the aluminum specimens, Figure 2, have diameters of 50 µm.

These two materials have dramatically different tensile properties. The fracture strength of the polycrystalline silicon, Figure 1, is around 5 GPa, near the generally-accepted value of the maximum tensile strength of a crystalline material, at about one-thirtieth of its Young's modulus, which is about 160 GPa. The behavior of the commercial CMOS aluminum films, Figure 2, was surprising. Pure aluminum film 1 µm thick, deposited at NIST by electron beam evaporation and tested previously by this technique, had a strength of about 125 MPa, consistent with its fine grain size, and an elongation of around 30 %. But the commercial CMOS material, containing 0.5 % copper, had low strength, around 70 MPa, and low elongation, around 1 %. The reasons for this behavior are under investigation.

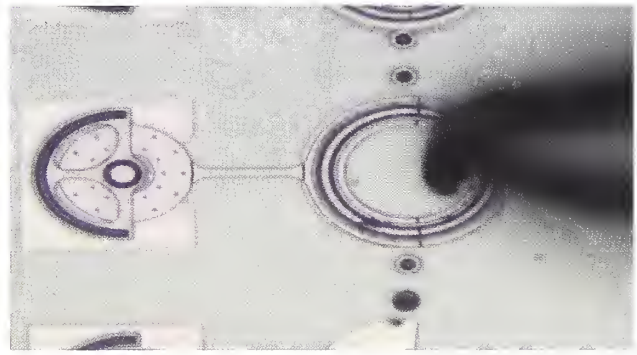


Figure 1. Polycrystalline silicon specimen made by Sandia National Laboratories, showing pull ring and swivel hub. Sandia pulls these specimens with a truncated conical diamond indenter. We use a tungsten loading pin. Similar results were obtained.

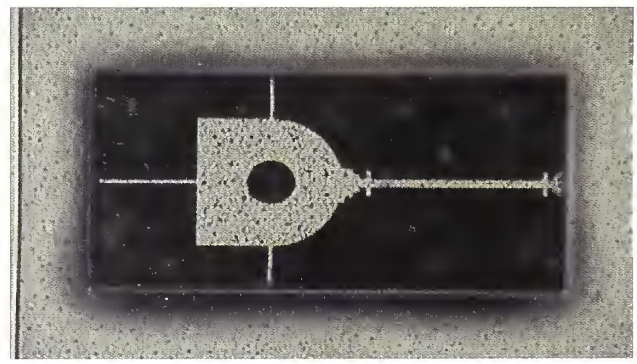


Figure 2. Tensile specimen in a polycrystalline aluminum interconnect layer from a commercial CMOS fabrication facility.

Contributors and Collaborators: Y.W. Cheng, J.D. McColskey, R.R. Keller (NIST)
Betty Yeung (Motorola, Tempe, AZ)

Electromigration and Stress Voiding in Interconnects

Robert R. Keller

Ever-improving performance of microelectronic devices is accomplished by scaling down the dimensions of metal interconnections. Generic, empirical lifetime models are insufficient for lifetime prediction since the roles of internal stresses and microstructure in such small structures are not thoroughly considered. This project addresses the mechanisms that control reliability in interconnections. Specific goals include studies of time-varying reliability degradation phenomena, localized stress measurement, and the role of local variations in microstructure in electromigration failure.

Background

Stress voiding (SV) and electromigration (EM) are failure phenomena that limit the reliability of narrow interconnects. They occur during thermal- and electric current-induced stressing, respectively. The end result is the formation and growth of hillocks and voids in the metal due to the development of severe mechanical stresses and stress gradients. Hillocks can lead to passivation cracking and short circuit failures, and voids to open circuit failures. Stresses result from differential thermal expansion among the metal, substrate and rigid passivation overlayer, or from atomic flux divergences due to non-uniform local diffusion during electrical stressing. Unless a more complete mechanistic understanding is developed, the impact of SV and EM is projected to worsen as the dimensions of interconnect structures continue to scale downward, and as new materials are introduced into interconnect architectures.

Electromigration testing of microelectronic interconnects is typically conducted via dc stressing. However, the vast majority of in-use conditions involves time-varying currents. Degradation mechanisms are not expected to be the same in these cases, based upon what is known about monotonic versus cyclic mechanical reliability in large-scale materials. We have therefore established an effort addressing alternating current-induced degradation of interconnects. Goals include characterization of failure processes and the influence of local variations in microstructure. We use electron microscopy to quantitatively characterize on a local scale the microstructures of films and narrow metallizations for interconnects. Electron backscatter diffraction, scanning electron microscopy (SEM), and transmission electron microscopy are the primary measurement techniques. Specifically, variations in grain orientations, surface damage morphology, dislocation configurations, and lattice parameters are measured and related to the observed reliability behavior. Results are interpreted in terms of damage accumulation models, and correlated to interconnect lifetimes.

Accomplishments

Progress on this project during FY01 was highlighted by R. Keller's nine month visit to Max-Planck-Institute for Metals Research in Stuttgart, Germany. We investigated the damage and lifetimes associated with cyclic stressing at high current density (> 5 MA/cm^2) of aluminum interconnects. Under such conditions, conventional electromigration processes are not expected to take place, due to times insufficient for significant diffusion to occur. Instead, we observed what appears to be damage due to thermomechanical fatigue induced by temperature amplitudes of roughly 50 to 100 K due to Joule heating. Such temperature changes induce a cyclic thermal strain of 0.1 to 0.2 % and corresponding stress of 70 to 140 MPa for aluminum lines on silicon substrates. The damage and lifetime behavior are consistent with those expected during fatigue testing of bulk metals. Namely, slip band upsets lead to surface roughening, and lifetimes follow published S-N (Wöhler) data for bulk aluminum. Figure 1 shows SEM images of the surfaces of a cyclically deformed Al film and an ac-stressed Al-1Si line; the damage morphology is very similar. We suggest that soft encapsulation materials may not suppress such damage as effectively as hard materials, and that fatigue may become a significant reliability issue for interconnects surrounded by polymeric materials.

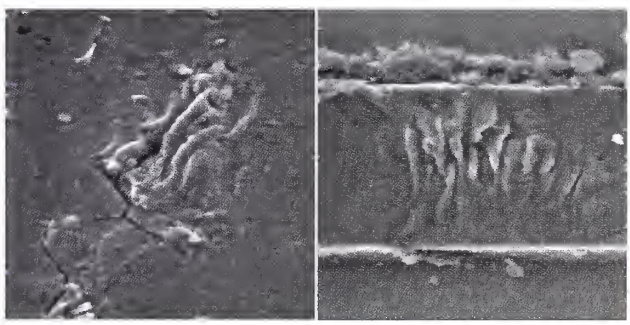


Figure 1. Al film on left subjected to 10^6 mechanical cycles at 10 Hz, strain amplitude of 0.8 %. Al-1Si line on right subjected to 10^7 current cycles at 100 Hz, current density of 10 MA/cm^2.

Contributors and Collaborators:

Fred Fickett (NIST)
Oliver Kraft, Reiner Mönig, Cynthia Volkert (Max-Planck-Institut für Metallforschung)

X-ray Methods for Materials Studies

Tom Siewert, Davor Balzar

We strive to correlate and explain macroscopic properties of technologically interesting materials by their underlying microstructure. We especially focus on x-ray diffraction studies of ferroelectric, optoelectronic, photovoltaic, semiconducting, and other materials relevant to the microelectronics and wireless communications industries. In particular, studies of microstructural properties, such as strain or stress, crystalline defects, and texture, complement the information obtained by the refinement and determination of short-range and long-range crystal structure in materials.

Background

Many macroscopic materials properties depend on the crystalline structure, internal stress, texture, and defect concentration. We develop methods to obtain this information from diffraction measurements, mostly for applications in microelectronics and wireless communications. Diffraction measurements are compared to measurements by other experimental techniques, such as atomic-force microscopy (AFM) and scanning electron microscopy (SEM). The properties are then correlated with other physical properties, typically dielectric, ferroelectric, magnetic, and electric.

Accomplishments

In this period, we focused on the development of methods for the characterization of combinatorial libraries of materials and the determination of residual stresses and strains for arbitrary crystalline symmetry. For speedy and automated characterization of combinatorial libraries, we developed a new instrument comprising a microfocus Cu-target x-ray source, *x-y-z* specimen stage with better than 0.5 mm accuracy, and an intrinsic-Ge solid-state detector. This detector permits both angle-dispersive and energy-dispersive measurements. As our first test, we studied a transparent conducting oxide (TCO) Cd-Sn-O one-dimensional combinatorial library. TCOs play a key role in a number of thin film optoelectronic devices including flat-panel displays, low-emission windows, photovoltaics, electrochromic devices, and anti-static coatings. The library is a glass strip, about 25 mm long, coated with the 600 nm thick Cd-Sn-O layer with a Cd/Sn composition gradient. First, we conducted an angle-dispersive high-resolution scan of the whole library, to identify the crystallographic phases that were present. Then, we rapidly performed spatially resolved energy-dispersive scans along the length. Energy-dispersive scans permitted a stationary sample and detector setup, which minimized sampling-volume positioning errors that were likely to occur during an angle-dispersive scan. Furthermore, the whole diffraction pattern was collected simultaneously, which allowed for quicker scans. Collected diffraction patterns were modeled by Rietveld refinement. Figure 1 shows plots of the refined lattice parameter as a function of position.

The parameter is a minimum at a position between 3 and 5 mm from the starting point. That area was carefully measured through a longer diffraction scan to identify possible changes in the stoichiometry and phase composition.

Another area of interest was the development of methods for accurate determination of the strain and stress state in materials of arbitrary crystal symmetry. The stress state influences many different materials properties, which are especially important in engineering and technological applications. We developed a novel approach to model diffraction line shifts caused by elastic residual or applied stresses in textured polycrystals. The model yields the complete strain and stress tensors as a function of crystallite orientations, as well as the average values of macroscopic strain and stress tensors. It is particularly suitable for introduction in Rietveld-refinement programs. The effects of sample symmetry are also included and conditions for strain invariance to both symmetries are discussed.

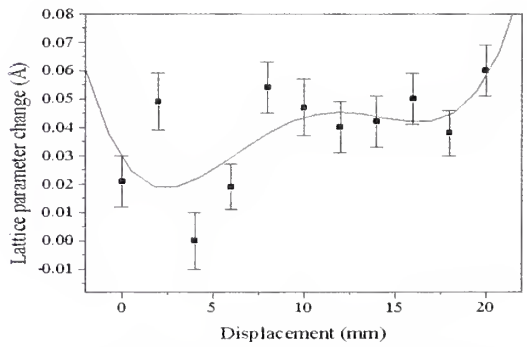


Figure 1. Lattice-parameter change as a function of position in a one-dimensional Cd-Sn-O combinatorial library.

Contributors and Collaborators: Nicolae C. Popa, Priscila Spagnol (NIST), John Perkins, Jeff Alleman, Joe del Cueto, Xiaonan Li, Tim Coutts, David Young, Phil Parilla, Brian Keyes, Lynn Gedvilas, Qi Wang, David Ginley (NREL), Dennis Readey, Chris Duncan (Colorado School of Mines), Renaud Stauber (University of Colorado)

Lead-free Solder Database

Members of the Materials Reliability Division are working with members of the Metallurgy Division, the Colorado School of Mines, and the National Electronics Manufacturing Initiative (NEMI) to expand a database on the properties of lead-free solders.

Tom Siewert

Technical Description

With product cycle time being slashed to keep up with consumer demand and competitive pressure, new electronic products are going directly from computer-aided design to full-scale production. The worldwide "green" movement in the electronics industry to replace lead-tin eutectic solders with lead-free solders creates a need for critical data on the industry's new lead-free solder compositions for these design and reliability models. In fact, the NEMI web site describes their view of the situation as "The NEMI Lead-free Assembly Project was launched in 1999 to help North American companies develop the capability to produce lead-free products by 2001, with an eye toward total elimation of lead by 2004. The goals and focus of the project were determined by the findings of NEMI's 1998 roadmap and an industry task force formed by NEMI to investigate process and material considerations of lead-free electronics assemblies." Their program plan is being implemented by the Lead-free Assembly Project. Project work is organized into five groups, including an Alloy Group whose mission is to select a lead-free alloy, pursue an industry standard, and gather data.

Development of our database started in April 2000 after we became members of the NEMI Lead-free Alloy Group, and learned that modelers and production engineers need more data before they can switch their production lines to lead-free solders. The long history in the use of current lead-based solders means that these data sets are quite complete and widely available. The modelers and production engineers need equally complete sets of data on the various lead-free alternatives, so they can make informed decisions for their production applications. Researchers are rapidly developing corresponding data on lead-free alloys, but the data are widely distributed among the various technical journals and proceedings. In addition, it is beginning to appear as though differences in test procedures (e.g. loading rates and dwell times) may make some of the data inconsistent from laboratory to laboratory. Finally, individual researchers may be repeating some of the work of others, while other critical data needs are being overlooked. Making the existing data more widely available will address all of these issues.

We have been requested to focus on the three lead-free compositions that seem to have the widest interest: near the tin-silver-copper eutectic (Sn-4Ag-0.8Cu), near the tin-silver eutectic (Sn-4Ag), and near the tin-copper eutectic (Sn-0.8Cu). We have added data for eutectic tin-lead composition (Sn-37Pb) for comparison purposes, and have added data for other lead-free alloys as we find them. We have not added data for electronic materials other than solder.

Accomplishments

Our team continues to work with this NEMI Lead-free Alloy Group to gather into a single database the existing physical and mechanical property data that have been developed by researchers around the world. The most recent version of the database (3.0) is posted on the Materials Reliability Division's web site at: http://www.boulder.nist.gov/div853/. In addition, our team is working with NEMI to refine a list of missing data (in order of ranked importance), with the list serving as a roadmap for research in lead-free solders. NIST and NEMI hosted a joint national workshop on these issues in February 2001, in conjunction with the TMS meeting in New Orleans.

We reported on the availability of the database in posters at the May 2001 ECTC meeting and May 2001 American Welding Society meeting, and received favorable comments from attendees at the poster sessions. Feedback from users of the database is guiding the addition of new data and we plan to critically evaluate more of the existing data each month.

Contributors and Collaborators:

David Smith (NIST)
Carol Handwerker, (Metallurgy Div., Chair of NEMI Lead-free Alloy Group)
Members of NEMI Lead-free Alloy Task Group
Juan Carlos Madeni, Steven Liu (Colorado School of Mines)

Materials for Wireless Communication

Today, wireless technologies constitute one of the most important growth areas in the global electronics industry. The current revolution in wireless communications would not have been possible without the discovery and development of oxide ceramics exhibiting the coincidence of high, temperature-independent resonant frequency with low dielectric loss. Technically important ceramic materials fall into two major dielectric categories: bulk ceramics for base station resonators/filter and those needed for low-power, miniaturized hand-held devices. Paramount in achieving smaller and lower cost devices are guidelines that facilitate the rational design of advanced materials to provide temperature stability, frequency, and size-reduction requirements for the next generation of devices for cellular, PCS, and many other niches of the wireless communications industry.

A collaboration between the Ceramics Division and the Electrical and Electronics Engineering Laboratory at NIST on ceramic materials for base station applications includes experimental determination of selected complex-oxide phase diagrams integrated with in-depth structural (x-rays, electrons, neutrons), crystal-chemical, spectroscopic, and dielectric property characterization. The objective of this multidisciplinary project is to determine the fundamental relationships between phase chemistry, crystal structure, and dielectric performance at wireless frequencies.

Another project in the Ceramics Division uses first-principles calculations to elucidate the roles of cation order-disorder and ferroelastic phenomena in determining the phase relations and physical properties of complex ceramic oxide systems. These calculations are used to predict cation ordering phenomena, physical properties, and how they vary with chemical composition. Critical experiments are performed to test the predictions.

Microstructural modeling and experimental studies are also underway to determine the dimensional changes in low-temperature-cofired ceramics used for portable communication devices. Processing models were identified by a large segment of industrial producers as being crucial to reducing the time currently needed to design and produce components. Wireless devices, often hand-held, are especially susceptible to mechanical damage; assuring the reliability of electronic products is especially critical for such devices. Work in the Materials Reliability Division includes development of noncontact acoustic metrology to characterize wireless materials in both thin-film and bulk form. Also, the mechanical properties of thin films are investigated using laser-ultrasonic methods to generate and detect surface acoustic waves. The elastic property information obtained will result in improved predictive modeling of film performance. In addition, resonant-ultrasonic techniques are applied to new piezoelectric materials for SAW devices, used extensively as oscillators in hand-held devices.

Researchers in the Polymers Division developed a new test method that permits dielectric measurements of film substrates at frequencies of 1 to10 GHz. The project is exploring the relationships between dielectric properties and structure in a variety of polymer resins, blends and composites, e.g., hybrid materials based on polymer resins and ferroelectric ceramics. Microstructural modeling and experimental studies are being undertaken to determine the effect of polarizability of the polymer matrix on the apparent dielectric permittivity of the composite. A need for models and measurement techniques has been identified by a large segment of industrial producers as being crucial in new designs and reducing the development time of new products. A consortium involving the National Center for Manufacturing Science and industry has been organized to help address these issues.

Contact Information: David T. Read (NIST)

Packaging Reliability

Develop methods to examine materials and interfaces in electronic packaging applications and elucidate the damage mechanisms. Our current focus is on advanced packaging structures and embedded passive materials using electron-beam moiré to measure mechanical strain, and thermal microscopy to measure heat flow and thermal properties on increasingly smaller size scales.

Andrew Slifka, Elizabeth Drexler

Technical Description

The microelectronics industry is moving rapidly toward higher density components of smaller size using less expensive materials. Examples are embedded and integrated passive components in printed circuit boards (PCBs), and another is the use of PCBs in various types of grid-array packages. These organic-based PCBs can have a large coefficient of thermal expansion (CTE) in comparison with many materials found in proximity. This CTE mismatch can reduce the reliability of electronic packaging systems by causing localized stress.

We are investigating the damage induced from CTE mismatches between organic materials, organics and metals, organics and ceramics, and organics, metals and ceramics to determine the initiation of damage and the ultimate failure mechanisms in these systems. Electron-beam moiré is employed to measure displacements and calculate mechanical strain due to thermomechanical loading. Thermal microscopy is used to measure changes in interfacial thermal resistance in order to detect the onset of thermomechanical damage at that interface before any surface manifestation is visible.

We are developing new measurement methods, both thermal and mechanical, using scanned-probe microscopy (SPM) in order to characterize packages at increasingly smaller size scales.

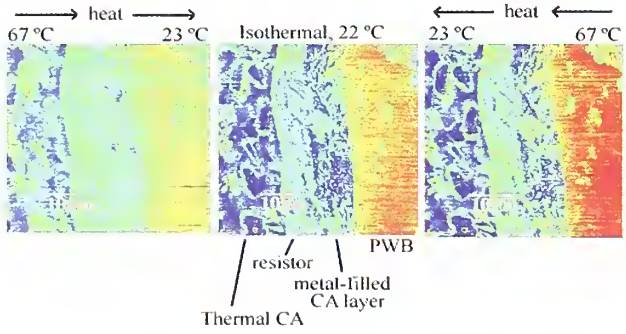


Figure 1. Scanning Thermal Microscopy images of an integral resistor from a U.S. manufacturer.

Accomplishments

Joule heating was utilized as the loading source in an experiment on an integral resistor. This approach localized the heating in the way it occurs in-service, and allowed us to validate thermal cycling as the accepted method to accelerate failure in electronic packaging materials.

We have made preliminary measurements, both thermal and mechanical, using SPM. We are acquiring a new SPM that will enable us to quantitatively measure the change in thermal resistance of interfaces with much smaller spatial resolution than is currently available with our infrared thermal microscope. Figure 1 shows a series of Scanning Thermal Microscopy images of an integral resistor sandwiched between printed wiring board and a thermally conductive adhesive. This SPM also offers the only opportunity for development of a scanning probe moiré technique, in conjunction with thermal SPM. Improved spatial resolution, such that displacements in submicrometer on-chip interconnects are quantifiable, is the motivation for developing this SPM moiré technique.

An electron-beam moiré test was conducted on an IBM PCGA (plastic column grid array) specimen to evaluate the elastic compliance of the solder column that connects the ceramic-encased die to the PCB substrate.

We conducted tests of integral and embedded resistors from two manufacturers using thermal microscopy and electron-beam moiré, as appropriate. Results were presented at quarterly meetings of the Advanced Embedded Passive Technology (AEPT) consortium.

Software was acquired and a program was written to control the electron beam of the new FE-SEM in order to perform lithography on moiré samples. The field-emission system has a smaller beam diameter, which will produce a smaller pitch for the specimen grating. Ultimately, this will provide increased resolution for displacement measurements. We are in the process of ascertaining the proper exposures for crossed line gratings using the new lithography system.

Contributors and Collaborators:

T. Andrew Winningham (University of Colorado, Boulder, CO)
Joe Kuczynski (IBM, Rochester, MN)
Virang Shah (MicroFab Technologies, Plano, TX)
Richard Snogren (SAS Circuits, Inc., Littleton, CO)

Combinatorial Methods

The Combinatorial Methods Program develops new measurement techniques and experimental strategies needed for rapid acquisition and analysis of physical and chemical data of materials by industrial and research communities. A multi-disciplinary team from the NIST Laboratories participates to address key mission driven objectives in this new field, including needed measurement infrastructure, expanded capability, standards and evaluated data.

Measurement tools and techniques are developed to prepare and characterize materials over a controlled range of physical and chemical properties on a miniaturized scale with high degree of automation and parallelization. Combinatorial approaches are used to validate measurement methods and predictive models when applied to small sample sizes. All aspects of the combinatorial process from sample "library" design and library preparation to high-throughput assay and analysis are integrated through the combinatorial informatics cycle for iterative refinement of measurements. The applicability of combinatorial methods to new materials and research problems is demonstrated to provide scientific credibility for this new R&D paradigm. One anticipated measure of the success of the program would be more efficient output of traditional NIST products of standard reference materials and evaluated data.

Through a set of cross-NIST collaborations in current research areas, we are working to establish the infrastructure that will serve as a basis for a broader effort in combinatorial research. A Combinatorial Methods Working Group (CMWG) actively discusses technical progress within NIST on combinatorial methods through regular meetings. The technical areas and activities of the CMWG are available in a brochure "Combinatorial Methods at NIST" (NISTIR 6730). Within MSEL, novel methods for combinatorial library preparation of polymer coatings have been designed to encompass variations of diverse physical and chemical properties, such as composition, coating thickness, processing temperature, surface texture and patterning. Vast amounts of data are generated in a few hours that promote our understanding of how these variables affect material properties, such as coatings wettability or phase miscibility. Additional focus areas for both organic and inorganic materials include multiphase materials, electronic materials, magnetic materials, biomaterials assay, and materials structure and properties characterization. State of the art on-line data analysis tools, process control methodology, and data archival methods are being developed as part of the program.

In order to promote communication and technology transfer with a wide range of industrial partners, an industry-National laboratories-university combinatorial consortium, the NIST Combinatorial Methods Center (NCMC) is being organized by MSEL. The NCMC will facilitate direct interactions on combinatorial measurement problems of broad industrial interest and efficient transfer of the methods developed to U.S. industry.

Contact Information: Vinod K. Tewary (NIST)

Modeling and Characterization of Combinatorial Libraries

A variety of measurement techniques are being developed and adapted for screening and characterization of combinatorial libraries of functional materials. Methods include x-ray diffraction, scanning acoustic microscopy, dynamic atomic force microscopy, thermal screening, and magneto-acoustic techniques. A theoretical model based upon the elastodynamic Green's functions and boundary-element analysis is being developed that would give the local acoustic response of the library.

Vinod K. Tewary

Technical Description

This year we have started a new program on modeling and characterization of combinatorial libraries of functional materials. A combinatorial library of materials is in the form of a thin film containing several (10^2-10^4) sites of new materials on a common substrate. A fundamental understanding of the physical properties of new materials requires knowledge of their elastic characteristics such as stress, elastic constants, and texture. Stress is a key factor that can influence mechanical, dielectric, thermodynamic, and optical properties of thin films. It limits the maximum thickness of epitaxial films, alters ferroelectric domain structure, the Curie temperature, and tunability of microwave devices such as resonators, filters, and phase shifters.

Our goal is to develop a mathematical model and measurement techniques for evaluating the structural, mechanical, thermal, and magnetic properties of libraries of new electronic, magnetic, and polymeric materials. The technical challenge lies in developing quantitative measurement techniques with micrometer spatial resolution, capable of rapidly scanning areas of a few square centimeters. New theories are required for modeling the elastic response of the library and to develop techniques for inversion of the measured values to determine the parameters characterizing the library.

Accomplishments

A variety of measurement techniques are being developed and adapted for library screening and characterization. A theoretical model based upon the elastodynamic Green's functions is being developed that would give the local acoustic response of the library. Measurement techniques include x-ray diffraction, scanning acoustic microscopy, dynamic atomic force microscopy, thermal screening, and magneto-acoustic techniques.

For speedy and automated x-ray characterization of combinatorial libraries, we developed a new instrument comprising a microfocus Cu-target x-ray source, x-y-z specimen stage with better than 0.5 mm accuracy, and an intrinsic-Ge solid-state detector. We studied a one-dimensional combinatorial library of transparent conducting oxide (TCO) Cd-Sn-O. TCOs play a key role in a number of thin film opto-electronic devices including flat-panel displays, low-emission windows, photovoltaics, and electrochromic devices. Details are given in a companion report.

We are developing a new method of measuring the magnetostriction of thin magnetic films on nonmagnetic substrates. Its primary advantage over conventional methods is that it can be performed on a local region of a blanket film on a wafer type substrate. Thus, it is well suited to scanning a combinatorial library of compositions that show promise of abnormally high (or low) magnetostrictive coefficients. Since application of the method to thin films is new, the FY01 program focused on a well known material (nickel) deposited on a silicon wafer. The figure shows the results obtained on a library in which the thickness of the Ni film varied over the face of the library. The qualitative features of this graph (including the thickness effect) can be explained by the existing model for the sensor.

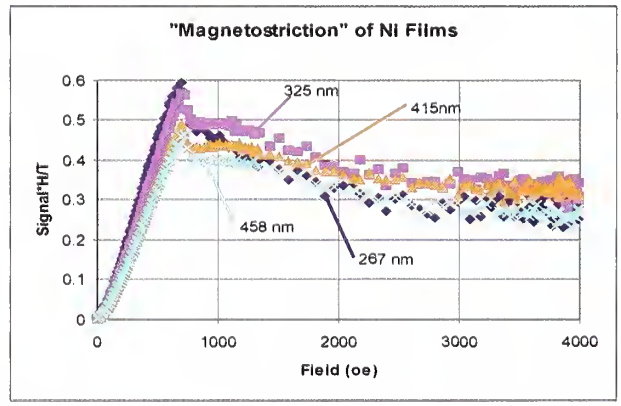


Figure 1. The ordinate is the measured signal amplitude from the sensor and is proportional to the magnetostriction coefficient of the film under the sensor divided by the magnetic field applied parallel to the film. This applied field is plotted as the abscissa.

Contributors and Collaborators: V.K. Tewary, G.A. Alers, D. Balzar, W.L. Johnson, A.J. Slifka (NIST) S.E. Russek (NIST, EEEL)

Ultrasonic Characterization of Materials

The program on Ultrasonic Characterization of Materials develops model-based methods for measuring those ultrasonic properties of a material that will permit characterization of microstructural features with size scales ranging from atomic arrangements to grains, reinforcements, and surface layers. Our goal is to convert these measurement methods into sensor systems suitable for in-service monitoring of material quality and serviceability as well as to provide physical property values to support the design of improved structures.

A primary focus of this program is the characterization of microelectronic structures such as interconnects and dielectric or magnetic layers as well as the microstructural features within structural alloys, composites, and engineered surfaces. The idea is to establish models that relate microstructure to measurable physical properties so that by measuring appropriate ultrasonic properties, the salient microstructural features can be inferred. For example, measurements of ultrasonic wave velocity and attenuation can be related to the elastic properties of microstructural features and to mechanical relaxations that reflect such dynamic processes as atomic diffusion or magnetic moment redistribution. These model-based measurements will enable industry to replace destructive microscope methods with nondestructive techniques for the microstructural characterizations needed to assure the quality of materials both for advanced electronics and for much larger structures.

The Ultrasonic Characterization of Materials Program is making significant contributions to measurement technology as well as to the modeling of material properties. We have worked with industry to commercialize noncontact ultrasonic transducers, waveform-based acoustic emission testing, magnetostrictive transduction, and nonlinear ultrasonics. Modeling advances include the following: Green's function methods for rapid solution of problems involving wave propagation in layered, anisotropic materials; data analysis procedures for deducing the elastic modulus tensor elements of anisotropic polycrystals; and techniques to separate internal friction values from ultrasonic attenuation measurements that are dominated by losses from scattering and beam diffraction. Current emphasis is directed toward measuring the elastic properties of thin films used in microelectronic devices.

Contact Information: George Alers (NIST)

Elastic-Stiffness Coefficients and Related Physical Properties

Our main goal is to understand, through measurements and modeling–theory, the elastic and related properties of solids that possess high scientific or technological interest. As required, we develop new measurement and modeling–theory methods.

Hassel Ledbetter, Sudook Kim

Technical Description

Our research emphasizes measurements and modeling–theory of elastic coefficients and related physical properties of metals, alloys, composites, ceramics, and the new high-Tc oxide superconductors. For many studies, the temperatures range between 295 and 4 K. The elastic coefficients, which relate deformation to stress, sustain our interest because they relate to fundamental solid-state phenomena: interatomic potentials, equations of state, and phonon spectra. Furthermore, thermodynamics links elastic coefficients with specific heat, thermal expansivity, atomic volume, the Debye temperature, the Grüneisen parameter, and many other fundamental properties, and with practical properties such as hardness.

Beside the elastic coefficients, we study sound velocities, internal friction, thermal expansivity, the Debye characteristic temperature, atomic volume, anharmonic properties (such as the Grüneisen parameter), creep, stress-strain behavior.

From the theoretical side, we can consider any physical property representable as a tensor. For example thermal conductivity, piezoelectricity, dielectric behavior, and so on.

Accomplishments

For the 2001 fiscal year, our accomplishments were extensive. This report's publications list gives titles of twenty-seven of our manuscripts either published in the archival literature, submitted for publication, or to be submitted for publication soon. Some of these continue last year's research efforts.

These studies focused on the following materials and properties:

Attenuation in composites
Cementite (Fe_3C)
Composites
  Cu/steel
  Diamond/Cu
  NbTi/Cu/epoxy
  NiTi/Al
  SiC_I/Al
  SiC_I/glass
  SiC_p/Ti
Covalent compounds
Diamond crystals
8-N Compounds
Galena (lead sulphide)
Gold-tin joining alloy
Langasite ($La_3Ga_5SiO_{14}$) crystal
Langatate ($La_3Ga_{5.5}Ta_{0.5}O_{14}$) crystal
Martensite (various studies)
Mullite/silliminate composite
Mullite/Al_2O_3 composites
Oxides
Oxide superconductors
Quartz crystal
Steel, low-carbon
Steel, Cu-precipitated
Steel, 4340
Tin/Silver joining alloy
Titanium diboride crystals (2 studies)
Tungsten-copper laminates

Our 2001-fiscal-year publication list includes numerous review papers.

Perhaps our most notable achievement this year was publishing eleven chapters to the Handbook of Elastic Properties of Solids, Liquids, and Gases, which will provide the standard elastic-property reference for many years. Also, to the forthcoming Encyclopedia of Materials, we contributed an invited review on solid-state lattice-vibrational properties.

Output

This report's publications list shows twenty-seven of our manuscripts that emerged during the 2001 fiscal year.

Contributors and Collaborators:

H. Ogi (Osaka University)
M. Dunn (University of Colorado)
P. Heyliger (Colorado State University)

Electromagnetic-Acoustic Resonance Methods

Ward Johnson

This project seeks to develop measurement and analysis techniques for determining the elastic and anelastic properties of materials using noncontacting electromagnetic excitation of resonant acoustic modes. The research includes development of transduction techniques employing tone-burst excitation, analysis of time-dependent waveforms involving superimposed resonances, and numerical and group-theoretical analysis of measured spectra for determining elastic constants, damping coefficients, and vibrational mode symmetries. The current focus is on using these techniques to characterize materials with electronic applications.

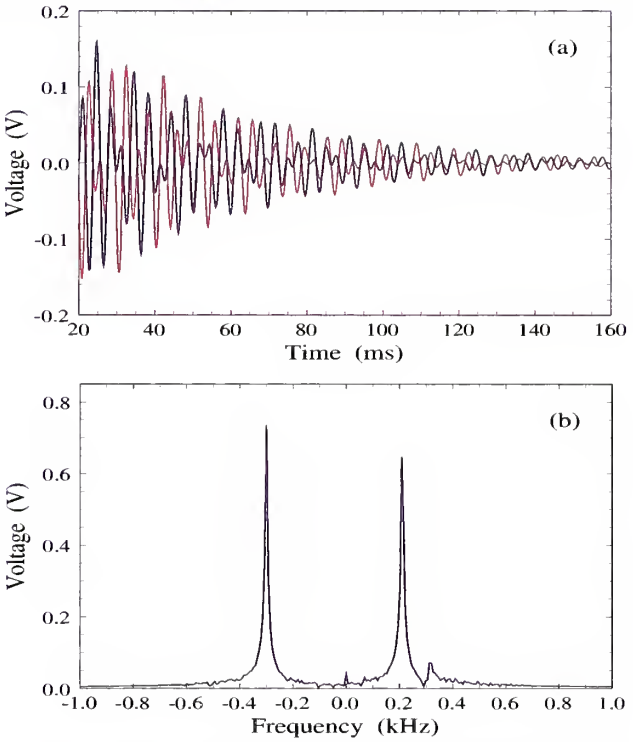
Technical Description

All conventional techniques for measuring elastic and anelastic properties of materials are in some respects limited in accuracy and ease of use. Methods that employ contacting transducers introduce errors because they alter the boundary conditions. Plane-wave techniques are severely limited with respect to measurements of damping (Q^{-1}) because of geometric spreading of the waves. Swept-frequency resonance techniques are limited by relatively long measurement times. Tone-burst resonance excitation techniques are limited by an inability to analyze complex waveforms when several resonant modes are simultaneously excited. This project seeks to overcome all these limitations by developing noncontacting tone-burst resonance methods in conjunction with variable-separable nonlinear least-squares fitting of waveforms and analysis of frequencies using the Ritz variational method.

This research effort is currently focused primarily on bulk piezoelectric materials for electronic oscillators, and thin films deposited on semiconducting substrates. Inductive piezoelectric techniques are employed with piezoelectric materials, and interdigital capacitive transduction techniques are being developed for thin-film materials.

Accomplishments

Complex waveforms arising from the simultaneous tone-burst excitation of several resonant modes have been acquired using phase-sensitive detection and analyzed using a variable-separable nonlinear least-squares fitting algorithm. An example of a signal from langatate and the corresponding magnitude of the FFT (referenced to the drive frequency) are shown at right. A variable-separable nonlinear least-squares fit (not shown) to superpositions of several decaying sinusoids yields resonant frequencies and corresponding Q^{-1} simultaneously over the entire bandwidth of the driving tone burst with resolutions that exceed that obtained from analysis in the frequency domain. This general least-squares approach has broad applicability to pulsed measurements of various types, including pulsed nuclear magnetic resonance.



a) In-phase and out-of-phase components of the decay of acoustic vibrations in langatate following excitation with a 1.097 MHz 1-ms tone burst. b) Corresponding FFT.

Computer algorithms employing the Ritz method have been developed for determining the elastic constants from measured resonant frequencies and fully characterizing the vibrational symmetries of a trigonal single-crystal cylinder. This provides an accurate and operationally simple method for determining the elastic properties, which are critical to the design and performance of electronic resonators. When applied to measurements obtained with noncontacting transduction, an extension of this analytical approach may enable, for the first time, the simultaneous determination of both elastic and piezoelectric constants.

Contributors and Collaborators: Bert Rust (NIST, Gaithersburg, MD)
Paul Heyliger (Colorado State University, Fort Collins, CO)

Ultrasonic Measurement of Residual Stress

The main objective of this program is to develop ultrasonic techniques and procedures that would allow measurement of residual stress in large structures, surface layers and thin films. The frequency (wavelength) of the waves used is expected to set the dimensional scale for the application and the use of several wave types will allow separation of the stress from other effects of the microstructure.

George Alers

Technical Description

Nondestructive measurement of the level of residual stress in large structures, coated parts and thin films is a long-term goal of the NDE community for predicting service-life and reliability. Since stress modifies the velocity of ultrasonic waves, it has often been suggested that an accurate measurement of the velocity of sound in a component could be used to infer or even measure the residual stress. Unfortunately, preferred orientation or texture in the grain structure of most materials introduces effects on the velocity of sound that mimic and often exceed the effects of the residual stress.

The objective of this project is to develop procedures for measuring several ultrasonic wave velocities so that the effects of stress and texture can be separated in the data analysis. A secondary objective is to use techniques that could ultimately be implemented on structures in the field or on parts at a manufacturing facility. To date, our program has focused on large metal structures where electromagnetic acoustic transducers (EMATs) could be used for launching and detecting the ultrasonic waves.

Accomplishments

During FY01, experimental apparatus and several types of EMATs were assembled and an extensive amount of very accurate data were collected on aluminum samples subjected to well defined applied stresses.

Figure 1 is a drawing of the apparatus developed for very accurately measuring the velocity of surface-skimming waves on thick samples or Lamb waves in thin plate samples. By recording the arrival time of a zero crossing in the middle of a tone-burst signal propagating between the transmitter and receiver EMATs as a function of the transducer separation distance, the phase velocity of the wave can be measured with an uncertainty of ±0.01 %. A similar uncertainty determines the accuracy with which one can determine the phase velocity as a function of the angle between the propagation direction and an axis fixed to the sample.

EMATs that generate and detect surface-skimming longitudinal and horizontally polarized shear waves in addition to surface acoustic waves are available for mounting in the apparatus shown in Figure 1. The results of measurements using all these transducers are being used for: (1) measuring the effects of plastic deformation in natural gas pipelines bent by large motions of the earth surrounding the pipeline; (2) determinating the errors introduced by magnetic contributions to the velocity of ultrasonic waves in steel; (3) assessing the errors caused by texture in the reference blocks used to calibrate the instruments used for ultrasonic inspections of welds; (4) measuring residual stress in the surface of thick aluminum plate.

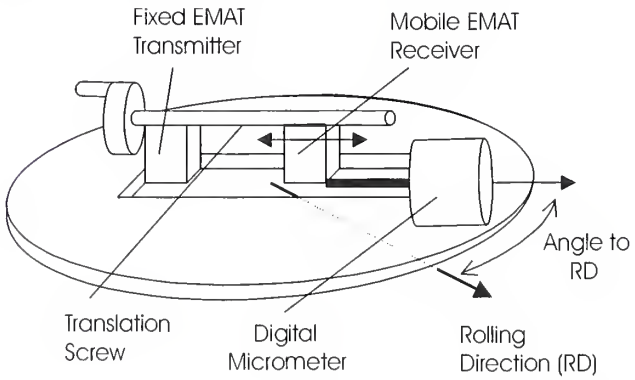


Figure 1. Apparatus for precisely measuring the phase velocity of surface-skimming ultrasonic waves.

Output

Two papers were submitted to the Review of Progress in QNDE, 2001 meeting at Bowdoin College, Brunswick, Maine, July 29-Aug. 3, 2001.

Contributors and Collaborators: G.A. Alers, J.D. McColskey and R.L. Santoyo (NIST)
M. Dunn (Univ. of Colo.), H. Haynes (Gas Technology Institute)
D. Kerr (Pacific Gas and Electric)
R.B. Thompson (Iowa State University), M. Warchol (Alcoa)

Modeling Acoustic Emission Signals for Waveform-Based Acoustic Emission

G.A. Alers, M.A. Hamstad

The major project objective is to develop a firm scientific base that will provide the underpinnings necessary to obtain the potential enhancements to AE technology due to an increased, high-sensitivity bandwidth. Current secondary objectives include: (1) developing for many users the missing element of modeling AE signals for all relevant geometries; (2) developing rational application of artificial intelligence technology to the real-world problems of reliable source identification and location of sources of AE signals.

Technical Description

Acoustic emission (AE) refers to the generation of propagating elastic displacement waves as a result of micro-sized releases of transient energy in a material. Monitoring these waves can provide fundamental information about the location and mechanism of the transient-energy release as well as the time/stress history of such releases. Often the energy release is due to local micro-damage processes as a response to applied stress. The technical approach, which is beyond that currently commercially offered for either resonant or waveform-based AE technology, is to successively examine different aspects of a multifaceted development of all the key components that are relevant to a wideband application of AE technology. These components include development of wideband high-sensitivity sensor/preamplifiers, high-speed digital recording data-gathering systems of wide dynamic range, finite-element modeling to predict near- and far-field displacement waves from relevant AE sources, wideband experimental AE displacement waveforms from typical sources in materials of interest, signal-processing techniques to accurately identify source types and their locations, and experimental studies of simulated propagation of AE waves. The scope in FY01 covered three related phases: (1) additional finite-element modeling of displacement signals (in the near and far fields) resulting from buried single- and multi-dipole point sources in laboratory-size samples as compared to large field-size samples; (2) computation of wavelet transform (WT) results for a variety of AE source types and source depths (relative to a plate's mid-plane); and (3) initial analysis of WT results to determine critical parameters available for extraction for AE source identification purposes.

Accomplishments

1. With the NIST developed finite element code,we have computed an extensive database of AE signals for 16 cases of single- and multi-dipole sources in an aluminum plate 4.7 mm thick. The plate has sufficient lateral dimensions so that plate edge reflections do not complicate the initial development of signal-processing techniques by superimposing on direct signal arrivals. From the signal database, a WT database has been computed from the out-of-plane displacement signals at three far-field distances from the AE sources.

2. Results from the WT database demonstrate that at three far-field propagation distances the WTs from a single source have recognizable similarities in the WT contour plots as modified by dispersion (See Figure 1). Note that amplitude of WT is shown by color; red is the highest amplitude.

3. Additional results from the WT database demonstrate promise of a WT-based technique for AE source identification. The source identification technique could be based on the intensity of the WT at key frequency-time data pairs (See Figure 2).

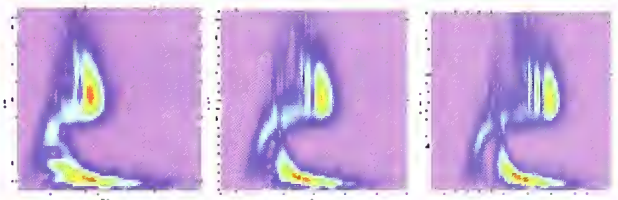


Figure 1. Wavelet transforms at three propagation distances (left to right: 60, 120, and 180 mm) showing frequency (0 to 1 MHz) versus time (0 to 80, 0 to 120, and 0 to 150 μs, respectively) for an in-plane dipole.

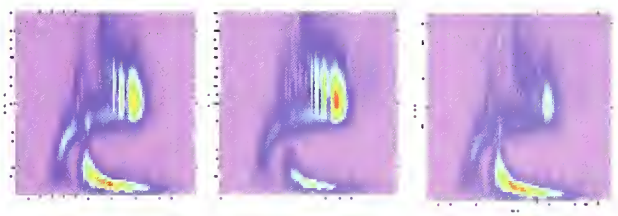


Figure 2. Wavelet transforms (for 180 mm propagation distance) showing frequency (0 to 1 MHz) versus time (0 to 150 μs) for three source types (left to right: in-plane dipole, out-of-plane dipole, and microcrack initiation).

Contributors and Collaborators: John Gary, Abbie O'Gallagher, David McColskey, Jen Newton (NIST) William Prosser (NASA Langley)

Green's Functions for Modeling Elastic Response of Electronic Materials

Vinod Tewary

A representation of the Green's function has been developed to model the elastostatic and elastodynamic characteristics of an anisotropic thin film on an anisotropic substrate. The elastodynamic model is used to calculate the dispersion of surface acoustic waves and Lamb waves in layered structures for micro electronic applications. A computationally efficient algorithm has been developed for inversion of the measured SAW and Lamb dispersion curves for determination of material parameters (elastic constants, density, and thickness) of thin films on anisotropic substrates.

Technical Description

Green's functions give the response of a solid to a probe. The Green's function provides a powerful tool for modeling the elastic response of anisotropic solids that is useful in interpreting measurements of their elastic characteristics. The elastostatic Green's functions are used for time-independent problems, and the elastodynamic Green's functions are used for modeling the propagation of elastic waves.

We calculate the elastodynamic Green's functions by using a delta-function representation in slowness space that we had developed earlier. This representation has been found to be computationally very efficient. In this representation, the Green's function at position vector **x** at time t is written as

$$\mathbf{G}(\mathbf{x},t) = \int \mathbf{g}(\mathbf{q})\, \delta\,[\mathbf{q}.\mathbf{x}-t]\, d\mathbf{q}, \qquad (1)$$

$$\mathbf{g}(\mathbf{q}) = \mathrm{Lim}_{\varepsilon\to+0}\, \mathrm{Im}\,[\Lambda(\mathbf{q}) - (1-i\varepsilon)\rho\mathbf{I}]^{-1}, \qquad (2)$$

$$\Lambda_{ij}(\mathbf{q}) = c_{ikjl}\, q_k\, q_l \qquad (3)$$

where Λ is the Christoffel matrix in slowness space, ρ is the density of the solid, **I** is the identity matrix, **q** is a vector in the slowness space, and the integral is over the entire vector space of **q**.

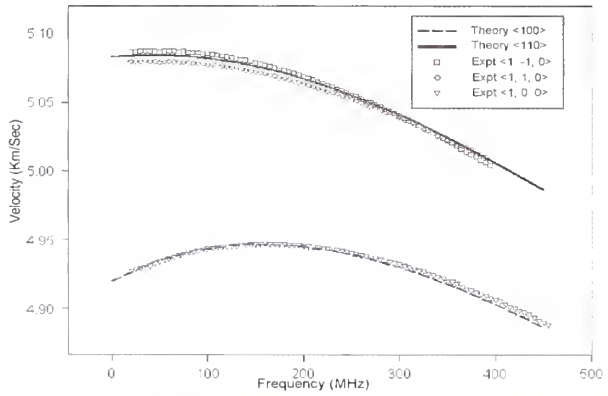


Figure 1. SAW dispersion curves in TiN/Si.

Accomplishments

We have developed a computationally efficient algorithm for inversion of the measured SAW and Lamb dispersion curves for determination of the material parameters of anisotropic films on anisotropic substrates. We have applied this method to estimate the elastic constants of thin films of TiN on single crystal Si from measured SAW dispersion curves (see Figure 1) in three directions (1,0,0), (1,1,0), (1,-1,0). Our calculations of Lamb dispersion in thin layered solids, which have not yet been verified experimentally, show that even at relatively low frequencies, elastic wave propagation can be sensitive to the material properties of the film. As shown in Figure 2, we find interesting transition effects where the symmetric mode crosses the transversely polarized mode.

Output

1. A joint paper on SAW methods to determine anisotropic elastic properties of thin films has been accepted for publication, and a paper on the elasto-dynamic Green's functions for SAW in thin anisotropic films of electronic materials has been sent for publication.
2. A computer program for determination of the material parameters of an anisotropic film on an anisotropic substrate from the measured SAW and Lamb wave dispersion curves is ready.

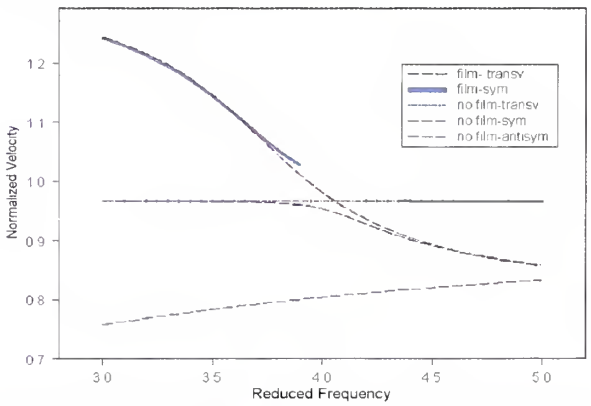


Figure 2. Dispersion of Lamb waves in TiN/Si.

Contributors and Collaborators: V.K. Tewary, D.C. Hurley (NIST)
L. Bartolo (Kent State University), Adam Powell (MIT)
J.R. Berger (Colorado School of Mines).

Elastic Properties of Thin Films Using Surface Acoustic Waves

We are developing noncontact, nondestructive tools to measure the mechanical properties of thin films. Our approach utilizes laser-ultrasonic methods to determine the frequency-dependent velocity of surface acoustic waves. The elastic-modulus information provided by our methods will aid in developing processes for new materials. Furthermore, such mechanical-property information is needed to predict the reliability and performance of new thin-film components.

Donna Hurley

Technical Description

Industrial uses of thin films range from providing specialized physical properties for device applications to giving ordinary materials extraordinary resistance to wear or corrosion. However, successful development of a film requires an understanding of its mechanical properties. Such knowledge is needed to estimate residual stresses, to predict component reliability, and to determine if there is good adhesion to the substrate. Yet current methods are generally limited to destructive tests or even "try it and see." We are developing nondestructive methods to quantify thin-film mechanical properties. Our goal is to relate measurable properties like elastic modulus, residual stress, or adhesion to factors like component lifetime.

The experimental approach involves surface acoustic waves (SAWs). SAWs are well suited for our purposes, since their energy is concentrated near the surface but the energy decay away from the surface depends on wavelength. Hence the SAW velocity depends on wavelength relative to the film thickness. To obtain elastic-property information, we measure the SAW phase velocity over a broad frequency range (dispersion relation). This is then compared to predictions of an analytical model in order to determine quantitative values for the elastic moduli.

Accomplishments

The experimental and theoretical methods created last year were refined this year. The frequency response of the detection system was improved by changing to a point focus in the Michelson interferometer. As a result, the highest detected SAW frequency increased from 220 to 400 MHz. This improvement means that thinner films can be more readily examined and that more information can be obtained for thicker films. We also reduced the laser energy needed for SAW generation, making the method truly nondestructive for many materials. Development of a new inversion algorithm to analyze our measurements was also completed this year. The inversion method uses the Green's function for SAW propagation in elastically anistotropic layered systems and enables us to determine a film's elastic properties from the dispersion relations.

We demonstrated the validity of our methods with two model systems. An elastically isotropic aluminum film 1093 nm thick on a fused silica substrate represented the simplest test case. We obtained $E_{Al} = 68.6 \pm 0.2$ GPa for Young's modulus of the film, in excellent agreement with literature values for bulk aluminum (67-71 GPa). The second system was a molybdenum film 288 nm thick on a silicon wafer that required anisotropic analysis. We got $E_{Mo} = 307 \pm 4$ GPa for Young's modulus and $\nu_{Mo} = 0.297 \pm 0.002$ for Poisson's ratio of the molybdenum film. Poisson's ratio was in excellent agreement with literature values (0.292-0.301). Young's modulus was 4-6 % lower than bulk values (319-328 GPa), but was consistent with previous film data. We are also working to compare our results with those using nanoindentation methods at NIST-Gaithersburg, and to compare nanoindentation and SAW measurements between NIST and BAM (Berlin).

We applied our methods to a series of titanium nitride films deposited on single-crystal silicon wafers. The deposition conditions meant that the films were elastically anisotropic, as were the substrates. Several different dispersion relations were obtained for each sample and then analyzed with our fully anisotropic inversion technique. Values obtained for the elastic moduli c_{11} and c_{13} and the thickness d_{SAW} of the films are given below. We measured the film thicknesses d_{SEM} destructively using scanning electron microscopy (SEM) and found good agreement with the SAW results. Wafer bending methods were used to determine the compressive residual stress σ in the films (below). A complete understanding of the observed trends between the elastic moduli, thickness, and stress requires further experimental investigation.

#	σ (GPa)	c_{11} (GPa)	c_{13} (GPa)	d_{SEM} (nm)	d_{SAW} (nm)
1	5.4	456±2	183±8	287±27	279±24
2	3.5	467±20	154±17	348±30	331±31
3	2.9	468±10	121±16	793±135	803±17
4	1.8	479±7	121±6	949±150	951±70
5	0.5	510±2	92±1	3330±242	3721±139

Results of SAW experiments for titanium nitride films.

Contributors and Collaborators: D.C. Hurley, V.K. Tewary (NIST)
D.T. Smith (NIST, Ceramics Division)
A. J. Richards CSIRO (Australia)

Atomic Force Acoustic Microscopy

Donna Hurley

We are developing in-situ *measurement techniques to probe mechanical properties on the nanoscale. Our methods involve dynamic excitation of an atomic force microscope's cantilever at ultrasonic frequencies. The mechanical-property information obtained with this method will prove valuable for a range of applications involving thin films or small-scale structures. For example, AFAM tools may serve as a tool to monitor the cryopreservation process to improve viability rates.*

Technical Description

Ever-decreasing length scales in many fields of technology present a serious challenge for materials characterization. New nondestructive tools must be developed to accommodate submicrometer dimensions. Specifically, determination of nanoscale mechanical properties is needed in many applications, particularly microelectronics. Knowledge of mechanical properties like elastic modulus and interfacial quality (such as strain and adhesion) is critical to successful development of film materials and device assemblies. Likewise, nanoscale mechanical information could provide an assay tool for combinatorial materials discovery.

Biotechnological applications could also benefit. For instance, nanoscale mechanical information could assess the reliability of biocompatible coatings and tissue scaffolding. Another example involves tissue cryopreservation. Although the ability to cryopreserve tissue underpins much medical biotechnology, success is limited by understanding of the cryopreservation process. By sensing mechanical-property variations, nanoscale elastic techniques might differentiate between crystalline and noncrystalline ice formation and cell survival. In this way, such tools would help industry to better understand and optimize the cryopreservation process.

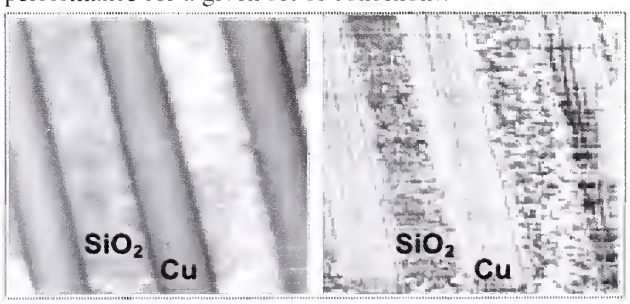
To meet these needs, we are developing tools that exploit the spatial resolution of atomic force microscopy (AFM). Although standard AFM measures topography, other new techniques sense a sample's elastic properties. One promising approach, called AFAM (atomic force acoustic microscopy), involves vibrating the cantilever at ultrasonic frequencies (~0.1-3 MHz) to excite mechanical resonances. The resonant frequencies shift as the tip comes in contact with a sample. By measuring the resonant frequencies under both free-space and surface-coupled conditions, quantitative information about the sample's elastic properties can be extracted. The small tip diameter enables *in-situ* measurements with nanoscale spatial resolution. Furthermore, AFAM promises 2D images of mechanical-property information.

Accomplishments

This year we developed experimental AFAM techniques using the apparatus created last year. Qualitative images were obtained by measuring the cantilever's vibration amplitude as the tip was scanned across the sample at a fixed excitation frequency. Such images of relative elasticity may provide valuable information about elastic stiffness variations among different sample regions.

We also worked to obtain quantitative AFAM information by analyzing shifts in the resonant frequencies. We investigated measurement issues like tip shape and wear and the need for reference and calibration samples. We checked our methods with an Al film 1μm thick on SiO_2 and a Nb film 0.3 μm thick on Si. With AFAM we got 101-106 GPa for Young's modulus of the Nb film, in good agreement with literature values of 100-110 GPa for bulk Nb. We measured values of 61-65 GPa for Young's modulus of the Al film. These are slightly lower than literature values of 67-71 GPa for bulk Al and 68.6 ± 0.2 GPa obtained on the same film with other methods.

To refine analysis and resolve such discrepancies, finite-element models are being developed through a university collaboration. Our goal is to understand the AFAM response of cantilevers with nonuniform shape and to probe the complex tip-sample interaction. In this way, experimental variables can be chosen to optimize performance for a given set of conditions.



Images of a Cu/SiO_2 microelectronic structure. Left: AFM topography image (50 nm full height). Right: AFAM image of relative elasticity. Images are 2 μm x 2 μm.

Contributors and Collaborators:

D.C. Hurley, P. Rice, M. Varney (NIST)
J.A. Turner (University of Nebraska-Lincoln)

Metals Characterization

Engineering design depends on the specification of the properties of the materials that are used. Equally important, manufacturers and their suppliers need to agree on how these properties should be measured. The MSEL Metals Characterization Program, centered within the Metallurgy and the Materials Reliability Divisions, spans the measurement spectrum from the innovative use of state-of-the-art measurement systems, to leadership in the development of standardized test procedures and traceability protocols, to the development and certification of Standard Reference Materials (SRMs).

The NIST effort in metals characterization has a strong emphasis on electron microscopy, which is capable of revealing microstructures within modern nanoscale materials and atomic-resolution imaging and compositional mapping of complex crystal phases with novel electronic properties. The MSEL microscopy facility consists of two high-resolution transmission electron microscopes (TEM) and a high-resolution field-emission scanning electron microscope (FE-SEM) capable of resolving features down to 1.5 nm. Novel experimental techniques using these instruments have been developed to study the mechanical properties of multilayered and nano-scaled materials.

The Metals Characterization Program is contributing to the development of test method standards through committee leadership roles in standards development organizations such as ASTM and ISO. In many cases, industry also depends on measurements that can be traced to NIST Standard Reference Materials (SRMs). This program generates the following SRMs for several quite different types of measurements.

- Hardness of Metallic Materials (Metallurgy Division): Hardness is the primary test measurement used to determine and specify the mechanical properties of metal products. The hardness standardization project is providing industry with primary transfer standards for the Rockwell hardness and Vickers and Knoop microhardness scales. These SRM test block standards are used for the periodic calibration of hardness testing machines.
- Magnetic Properties (Metallurgy Division): The need for reliable magnetic measurements is becoming increasingly acute because of new technologies involving magnetic phenomena in data storage and microelectronics. Such measurements require calibration of magnetometers using certified magnetic standards in several different shapes and magnetic strengths, and with a wide range in magnetic character. These standards are now being produced under this program.
- Coating Thickness (Metallurgy Division): Coating thickness standards are produced by electrodeposition and are widely used for calibration of coating-thickness measuring instruments. SRM coupons are produced with a wide range of thicknesses, and are bar coded to allow analysis of degradation and life expectancy when the standards are returned for verification.
- Charpy Impact (Materials Reliability Division): The Charpy impact machine verification project provides rapid, accurate assessment of test data generated by our customers using SRM Charpy standards, and, where merited, certifies the conformance of Charpy impact test machines to ASTM Standard E 23. Participation in ISO Committee TC 164, assures that specimens and procedures are compatible with international standards.

In addition to the SRM activities above, NIST (Materials Reliability Division) provides assistance to the Bureau of Reclamation (BOR) on metallurgical issues that arise during maintenance, inspection, and failure assessment of dams and water conveyance infrastructure projects. NIST advice and data provide BOR engineers with an independent check of other input.

Contact Information: Tom Siewert (NIST)

Charpy Impact Machine Verification

Daniel P. Vigliotti

1. Provide rapid, accurate assessment of test data, and where merited, certify the conformance of Charpy impact test machines to ASTM Standard E 23.

2. Interact with the ASTM Committee responsible for the Charpy impact standard, to improve the service to the customers and reduce the scatter in the data, and to meet the needs of industry.

3. Participate in the activity in ISO Committee TC 164, so our specimens and procedures remain compatible with the associated international standards and other regional standards.

Technical Description

The Charpy impact test uses a swinging hammer to assess the resistance of a material to brittle fracture. The absorbed energy is measured from a calibrated scale, encoder, and/or an instrumented striker. The low cost and simple configuration of the test have made it a common requirement in codes for metals used in critical structures such as pressure vessels and bridges. NIST provides highly characterized standard reference materials (SRMs) to machine owners and independent calibration services, then evaluates the results of tests of these specimens on their impact machines. Owners of machines that meet the requirements of ASTM Standard E 23 are given a letter of conformance, while owners of nonconforming machines are given recommendations on corrective actions.

Our special facilities include the three master Charpy impact machines (all roughly 300 J capacity). These three machines are used to establish certified values for the NIST reference materials sold through the Standard Reference Materials Program Office. This project is handled jointly by the Standard Reference Materials Program (Office of Measurement Services), which oversees the administrative aspects of the program, and the Materials Reliability Division, which handles the technical and certification aspects.

Accomplishments

We had about 1000 customers for this service in FY01, a gradual increase from the customer base of a few years ago. The great majority of these machines were within tolerances required by ASTM Standard E 23. As usual, we found that many users took advantage of our support services, as indicated by over 720 faxes and 4000 phone calls. This year, we implemented a new procedure to contact every customer whose machine fails to meet the verification criteria. In this contact (by phone, mail, email, or fax), we suggest corrective measures. In our laboratory, we tested the 1785 specimens necessary to confirm that 14 new lots of reference specimens were suitable to go into the SRM inventory.

We are in the first year of a new, three-year test program that is collecting data on "International Master Batches" of Charpy impact verification specimens. A meaningful harmonization (equivalency) of Charpy V-notch standards around the world is unlikely until the reference materials used for the verification of impact machines in Europe, Japan, and the United States share a more common method of certification. The results of this test program will be used to evaluate the use of Master Specimens as a common control in the certification procedure for CVN verification specimens between the three National Measurement Institutes. It will also evaluate machine variables, offsets, uncertainty, and other factors relevant to the harmonization of our respective systems.

We have implemented several new features to improve our service. In response to customer requests, we have ordered custom calibration stickers and are integrating the printing of the machine identification on the stickers with a new, more efficient database program. In addition, a series of heat treating experiments is underway to reduce the scatter within the reference sets, allowing better resolution of problems in the machines of our customers. Finally, we are producing replicas of broken Charpy specimens (with characteristic damage marks) that can be used by our customers to diagnose wear and damage on their machines.

Chris McCowan serves as the Chairman and U.S. Delegate to ISO TC164 SC4 P, on pendulum impact. Chris McCowan also continues as the Chairman of ASTM Subcommittee E28.07 on impact testing, and Dan Vigliotti continues as the Chairman of the Task Group that oversees Standard E23, the main standard for Charpy impact testing and as recording secretary to E28. We continue to use these ASTM meetings as a forum to discuss the statistical trends from our customer evaluations (percentages of machines that meet the requirements and the distribution of data around the mean). The technical committee members have been quite pleased with our openness in sharing these data.

Contributors and Collaborators: Dan Vigliotti (Charpy Program Coordinator), Chris McCowan, Tom Siewert, James Alcorn, Brian Marsh, Emma Nicoletti, Nicole Neumeyer (NIST)
Members of ASTM Subcommittee E 28.07

Service to Bureau of Reclamation

Provide assistance to the Bureau of Reclamation (BOR) on metallurgical issues that arise during the maintenance and inspection of dams and water conveyance infrastructure projects in the western United States. Typically, our advice regarding a particular issue is used by the BOR engineers as an independent check of other opinions they gather.

Christopher N. McCowan

The physical infrastructure of the United States contains diverse elements, and the material issues become even more complex as these structures age. To support their federally maintained infrastructures, the Bureau of Reclamation (BOR), within the Department of the Interior, relies on their Technical Service Center. When particularly complex problems call for additional expertise, the Center recruits experts from other government agencies and from the private sector. Here, we describe the interaction between BOR and NIST-Boulder on several recent materials issues.

Over the past several years, we have studied the failures of pre-stressing wires on pipes in the BOR's Central Arizona Project (CAP). Our examination of failed wires from the reinforced pipe, Figure 1, indicated that stress corrosion cracking initiated at pre-existing flaws in the wire, leading to the fracture of individual wires. Eventually, multiple wire failures severely degraded the structural integrity of the siphons, which convey water under roads and rivers in the CAP system. Our study of these failures contributed to a change in the type of pipes considered as options for these applications, and to the successful mediation of the issues in a lawsuit.

Another example of the NIST-BOR interaction for this year included the evaluation of admiralty brass tubes that failed on an air cooler at the Colorado Big Thompson Project, Figure 2. Our observations indicated that the failures of the brass cooling tubes were likely due to stress corrosion cracking. The cracking initiated where the tubing was rolled (expanded) into the tubesheet to make a seal. Measurements of the tubing thickness in the tubesheet indicate that the tubes may have been over-rolled. Over-rolling is known to produce high residual stresses at the back of the tubesheet that can drive circumferential stress corrosion cracking. We also performed and interpreted Charpy impact tests of failed connection bolts from Hoover Dam. We found that the failed bolts had very low toughness, while a potential replacement material looked quite good.

This interaction between the BOR and NIST has continued for over 8 years, and has led to a sharing of expertise and increased public safety.

Figure 1. A 6.4 m diameter siphon from the CAP. This section was excavated for inspection in about 1990 to help determine the extent of distress in the CAP system.

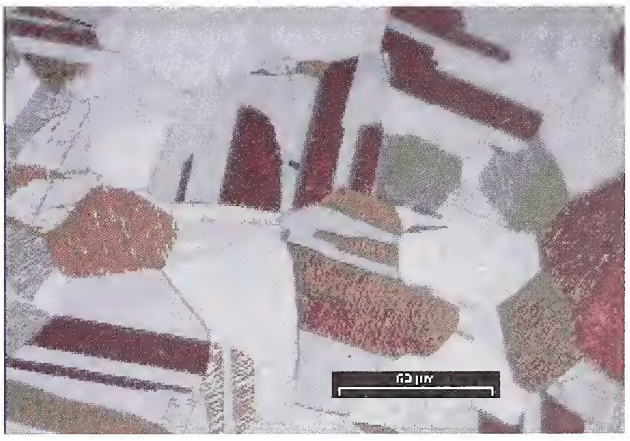


Figure 2. Admiralty brass sample showing equiaxed and twinned microstructure. The grain size in the tubing was not uniform.

Contributors and Collaborators: Chris McCowan, Tom Siewert, Dan Vigliotti (NIST)
Mike Peabody, Tom Johnson (Bureau of Reclamation)

Metals Processing

The Metals Processing Program applies NIST expertise in a wide range of disciplines, including thermodynamics, electrochemistry, fluid mechanics, diffusion, x-ray, and thermal analysis, to understand the processing steps which will lead to products having the desired form and properties, at an acceptable cost. Working with industries ranging as widely as automotive, aerospace, coating, and microelectronics, several important processing problems are being addressed including melting and solidification of welds, castings of single crystals, powder production and consolidation, and coating production by thermal spray and electrodeposition.

The increasingly competitive manufacturing environment fuels the search for new metal alloys as well as efficient processing techniques to fully realize their potential. The processing cycle can include many steps including a formation process such as casting or electrodeposition, a heat treatment process, a deformation process such as rolling or stamping, joining by welding, or coating to enhance surface properties. In each of these processes, the distribution of crystal phases, the grain structure, the alloy compositional segregation, and the defect structure are altered, with resulting changes in properties such as strength, ductility, corrosion resistance, and conductivity which form the basic rationale for the use of metals in industrial products. The following projects in the Metals Processing Program focus on measurements and predictive models needed by industry to design improved processing methods, provide better process control, develop improved alloy and coating properties, and reduce costs.

- Modeling of Solidification and Microstructure Development (Metallurgy Division): Models of alloy solidification, crystal growth processes and heat treatment are being developed to aid industry in designing production systems that increase product yield and performance.
- Processing-Structure-Property Data For Thermal Spray Coatings (Metallurgy Division): Coating reproducibility and reliability are addressed by the development and calibration of advanced sensors, applying these measurement tools to the control and characterization of TS coatings, and working with the Thermal Spray (TS) community to establish a coating processing-microstructure-property database.
- Weld Process Sensing, Modeling and Control (Materials Reliability Division): Advanced instrumentation and data analysis techniques are used to develop a better understanding of the underlying physics governing the arc welding process.
- High-energy X-ray Diffraction Studies (Materials Reliability Division): Investigates the use of high-energy x-ray diffraction as an alternate, nondestructive option to the conventional destructive methods for measuring physical properties.
- Tailored Metallic Powders (Metallurgy Division): Measurement techniques for the characterization of microengineered powders are developed to advance our understanding of the relationships among properties, processing, and microstructure.
- Electrodeposition of Aluminum Alloys (Metallurgy Division): Guidelines for the electrodeposition of aluminum-based alloys from low-temperature, low-vapor pressure non-aqueous electrolytes are being developed as an inexpensive method for producing homogeneous and fine-grained aluminum-based thin films for corrosion protection.
- Electrochemical Processing of Nanostructural Materials (Metallurgy Division): Electrochemical methods for the synthesis and characterization of nanoscale magnetoresistive device architectures are being examined with an emphasis on the use of surfactants and segregation phenomena in controlling homo- and hetero-epitaxial film growth.
- Reaction Path Analysis in Multicomponent Systems (Metallurgy Division): Costly experimental investigations of bonding and reaction processes involving interdiffusion at interfaces between metals, oxides, and vapors are supplanted by models, based on thermodynamic data, that predict the formation of transient phases and rates of reaction in complex multicomponent systems.

Metals Processing projects with an especially strong focus on areas which are of special interest to MSEL have evolved to become part of other program areas such as Materials for Microelectronics and Forming of Lightweight Materials. Because processing plays such a basic role in determining the properties and performance of metals, we expect this program to continue providing a foundation for advanced metals technologies.

Contact Information: Tom Siewert (NIST)

Research in Joining

Tim Quinn

Develop a better understanding of the underlying physics governing arc welding process through advanced instrumentation and data analysis techniques; develop simple, nonintrusive, and robust sensors that provide meaningful information about the status of the welding process; develop physics-based theoretical models of the process; and assist in the development of industrial standards for information exchange between intelligent components in a robotic arc welding cell.

Accomplishments

NIST personnel actively lead the development of a network standard for communication between components in the welding cell. The American Welding Society's A9 committee (chaired by NIST) is writing a standard (A9.4) to create a "plug and play" capability to integrate components in the welding cell. Currently, communication with a component in the welding cell is specific to that particular component, causing expensive, proprietary solutions to be generated for every weld cell's configuration. The A9.4 standard will allow any component in the weld cell to communicate with any other component using standard protocols and cabling.

A program was started to measure the mechanical properties of solders used to join electronic packages. The test apparatus was developed to measure the mechanical properties using samples of solder that are about the same size as are the solder balls used in grid-ball arrays (~250 mm). Equipment was purchased, and is currently being assembled to test these small specimens at low strain rates.

In conjunction with researchers at the Colorado School of Mines, NIST has developed a model of the wire in GMAW as it is being pulled through the conduit. An Euler contact model was developed to predict the amount of pulling force that is needed to pull the wire through the conduit as it goes around a curve. The model can predict the friction between the wire and the liner. Experiments were conducted using a linear actuator to smoothly pull the wire through a curve of a specific angle. Results agree with the model within a few percent.

Researchers at Ben Gurion University (Israel) and NIST have developed a coupled model of the arc, electrode and weld pool in gas metal arc welding (GMAW) by use of a commercial finite-element code. In the GMAW process, a metal electrode carries a large current (i.e. 300 A). An arc is created between the end of the electrode and the work piece in the inert shielding gas. As the electrode melts, droplets detach and travel into the molten welding pool. The important variables were identified through extensive sensitivity analyses. Convergence of the model was also tested. Experiments were conducted this year to verify the results of the finite-element model. Experiments are under way to measure the volume of the arc, to further test the model.

We studied the equilibrium temperature of the contact tube in gas metal arc welding, as the cross flow of cooling air was increased. Overheating of the contact tube is a common failure mechanism and water cooling brings reliability problems. Thus, air cooling seemed to be a natural solution to this problem. Figure 1 shows our data on the effectiveness of air cooling, and we exhibited these results in a poster at the May 2001 American Welding Exhibit.

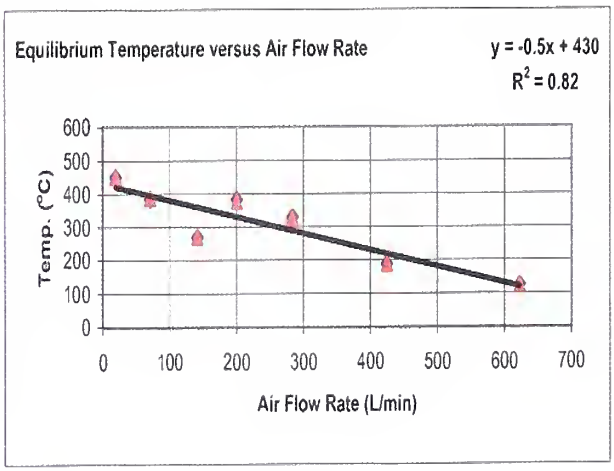


Figure 1. Reduction in equilibrium temperature of the contact tube as air cooling is increased.

Contributors and Collaborators: Tom Siewert (NIST, MSEL), Bill Rippey (NIST, MEL)
Toby Padilla, David Munoz (Colorado School of Mines)
Moti Szanto (Ben Gurion University, Israel)
Dave Farson (The Ohio State University)

Advanced Engine Materials

Ceramics and ceramic coatings play an enabling role as industry strives to meet demands requiring higher energy efficiencies, decreased emissions, extended engine life, and reduced warranty costs associated with diesel and turbine engines. Structural ceramics are already used in automotive and diesel engines as fuel pump and injector components, cam roller followers, water pump seals, and turbocharger rotors. These components provide increased wear and corrosion resistance and allow higher operating temperatures and thus improved engine performance. The use of ceramic coatings is increasing in both aircraft and land-based gas turbines, and in diesel engines. Most of the current use of ceramic coatings is associated with thermal barrier coatings – a thin ceramic layer deposited on metallic components to impart thermal resistance as well as resistance to environmental corrosion. The present historical trend of substantial increases in the introduction of ceramics and ceramic coatings in engines is expected to continue. However, the primary barriers to the widespread use of these materials are the cost and uncertainty associated with in-service mechanical reliability.

The primary objective of the Advanced Engine Materials Program is to provide measurement techniques, standards, basic data, and predictive models needed to develop and implement reliable and cost-effective materials for internal combustion and gas turbine engines. Research focuses on the development of test methods for the assessment of contact damage and wear, identification of machining-induced damage and its influence on mechanical properties, development of test methods and predictive models for evaluation of mechanical properties at elevated temperatures, development of techniques and reference materials for thermal conductivity measurements, and development of models for prediction of coating properties and performance. Reliability and precision of various measurement techniques and their suitability for standardization are assessed jointly with industrial partners, international measurement laboratories, and national and international standards organizations. The close working relationship developed between these organizations and NIST not only ensures the relevance of the research projects but also promotes an efficient and timely transfer of research information to industry for implementation.

Contact Information: Andrew Slifka (NIST)

Thermal Conductivity of Coatings

Develop steady-state measurement techniques and appropriate reference materials for thermal conductivity of ceramics and ceramic coatings used in advanced engines. This provides industry with calibration and transfer between transient and steady-state data, allowing understanding of the relationship between thermal performance and microstructure.

Andrew Slifka

Technical Description

Thermal-barrier coatings have many applications, but the primary use is in gas turbines and diesel engines, to allow higher operating temperatures and longer lifetimes for increase efficiency. New materials with designed microstructures for lowering thermal conductivity are being developed for coatings used in gas turbine engines, as well as nanoscale superlattices for thermal barriers. Industry uses a measurement of thermal diffusivity that is fast and generally reliable, but a bridge is needed to provide designers with thermal conductivity and to calibrate diffusivity measurement apparatus. To this end, we provide steady-state thermal conductivity measurement methods and data and develop appropriate reference materials.

Accomplishments

We are measuring ceramic coatings made by physical vapor deposition (PVD). These coatings are designed to lower thermal conductivity by controlling the coating microstructure. By adjusting process parameters, not only can the porosity be controlled, but the microstructure of the porosity as well. Figure 1 shows a PVD coating with low thermal conductivity due to a 3-level porous microstructure. Our measurements show that there is an optimal porosity mixture that reduces thermal conductivity by 50 %. This points material manufacturers in a direction to develop a new class of thermal barrier coating. The results of this study have been submitted for publication.

We are collaborating with the National Physical Laboratories (NPL) in the U.K. to study the relevant properties of Pyroceram 9606 as a standard reference material for thermal conductivity at high temperature. Earlier we made absolute thermal conductivity measurements of a lot of several specimens of U.S. Pyroceram 9606 for application as a NIST Reference Material (RM). As part of this collaboration we have now measured material provided by NPL in a collaborative effort to determine both the thermal conductivity and the consistency of that material and, ultimately, to provide an international reference material for conductivity in this important high temperature range. Figure 2 shows results from a measurement of material provided by NPL compared with measurements of our earlier material. The error bars show the 5 % uncertainty of the measurement method and the agreement between the two lots of material.

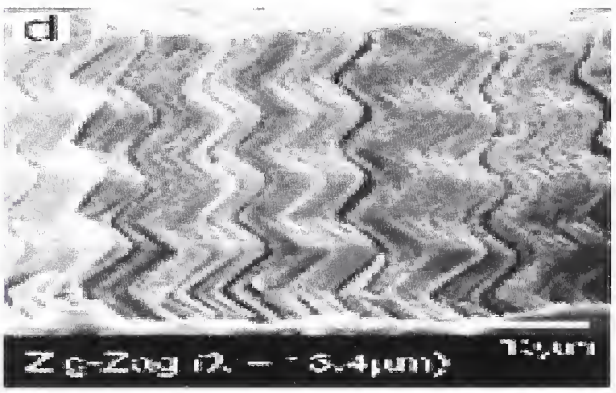


Figure 1. Ceramic coating made by PVD that exhibits low thermal conductivity due to a controlled porous microstructure

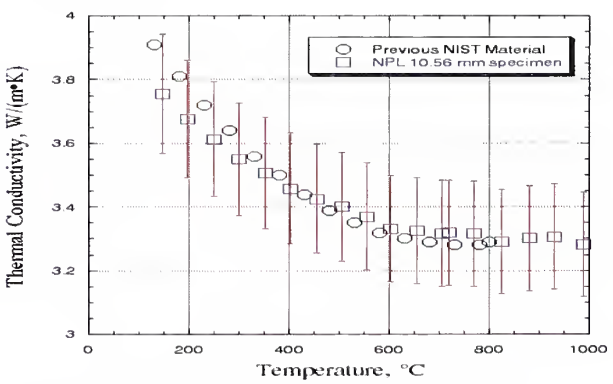


Figure 2. Comparison of thermal conductivity of Pyroceram 9606 from two different sources.

Contributors and Collaborators:

Dave Salmon (National Physical Laboratories, U.K.)
Derek Hass (University of Virginia)
H.N.G. Wadley (University of Virginia)

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Materials Reliability Division

Chief

Fred R. Fickett

Phone: 303-497-3785

E-mail: fickett@boulder.nist.gov

Deputy Chief

Thomas A. Siewert

Phone: 303-497-3523

E-mail: siewert@boulder.nist.gov

Group Leaders

Microscale Measurements

Fred R. Fickett

Phone: 303-497-3785

E-mail: fickett@boulder.nist.gov

Microstructure Sensing

George A. Alers

Phone: 303-497-7899

E-mail: alers @boulder.nist.gov

Process Sensing & Modeling

Thomas A. Siewert

Phone: 303-497-3523

E-mail: siewert@boulder.nist.gov

Research Staff

Alers, George
alers@boulder.nist.gov
- Nondestructive evaluation
- Electromagnetic acoustic transducers
- Process monitoring

Cheng, Yi-Wen
cheng@boulder.nist.gov
- Deformation and strain
- Scanning electron microscopy
- Microstructural characterization

Drexler, Elizabeth
drexler@boulder.nist.gov
- Electron-beam moiré
- Electronic packaging
- Micromechanical property measurement

Fickett, Fred
fickett@boulder.nist.gov
- Materials evaluation
- Magnetic sensors
- Scanned probe microscopy

Hamstad, Marvin
hamstad@boulder.nist.gov
- Acoustic emission
- Composite materials
- Nondestructive Evaluation

Hurley, Donna
hurley@boulder.nist.gov
- Ultrasonic measurements
- Laser ultrasonics
- Elastic properties of thin films

Johnson, Ward
wjohnson@boulder.nist.gov
- Ultrasonic measurements
- Internal friction
- Brillouin scattering

Keller, Robert
keller@boulder.nist.gov
- Materials science
- Electron microscopy
- Mechanical behavior

Kim, Sudook
sak430@boulder.nist.gov
- Elastic properties
- Low-temperature physical properties
- Ultrasonic measurements

Kumon, Ronald
kumon@boulder.nist.gov
- Nonlinear and microscale acoustics
- Surface acoustic waves
- Acoustoelastic effects

Ledbetter, Hassel
ledbetter@boulder.nist.gov
- Thermodynamic properties of solids
- Theory and measurement of elastic constants
- Phase-stability theory

McColskey, J. David
mccolske@boulder.nist.gov
- Crack propagation
- Composite materials
- Mechanical testing

McCowan, Christopher
mccowan@boulder.nist.gov
- Charpy impact testing
- Metallography and fractography
- Failure analysis

Quinn, Timothy
quinn@boulder.nist.gov
- Process modeling
- Welding automation
- Solder properties

Read, David
read@boulder.nist.gov
- Electronic packaging
- Elastic-plastic fracture mechanics
- Mechanical behavior of thin films

Santoyo, Ray
santoy@boulder.nist.gov
- Instrumentation
- Data collection and reduction
- Computer support

Siewert, Thomas
siewert@boulder.nist.gov
- Standard reference materials
- X-ray sensing and diffraction
- Weld sensing

Slifka, Andrew
slifka@boulder.nist.gov
- Thermal conductivity
- Thermal barrier coatings
- Surface characterization

Tewary, Vinod
tewary@boulder.nist.gov
- Solid state physics
- Green's function methods
- Elastic wave propagation

Vigliotti, Daniel
vigliotti@boulder .nist.gov
- Charpy impact testing
- Standard reference materials
- Instrumentation

Materials Reliability Division
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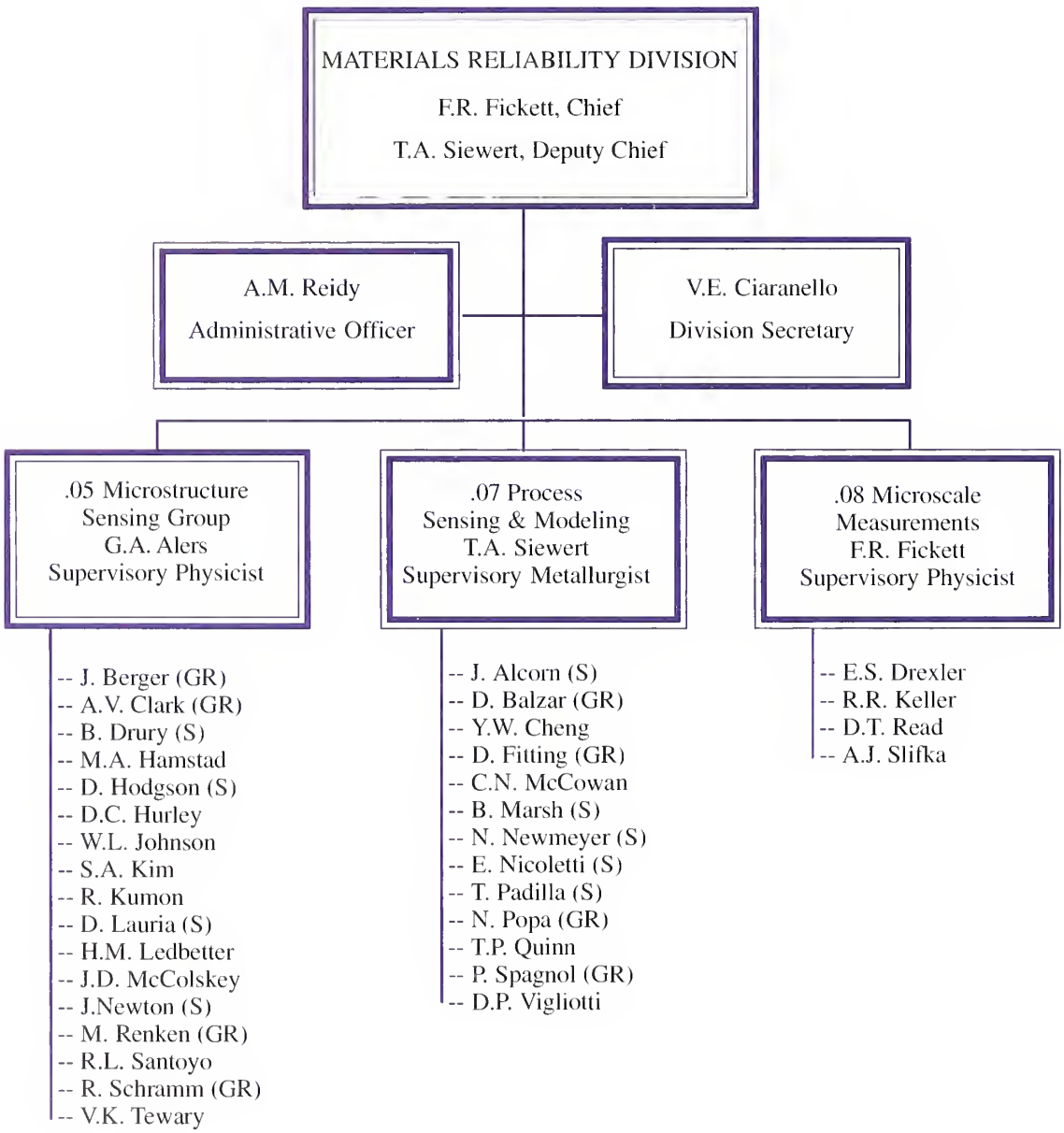
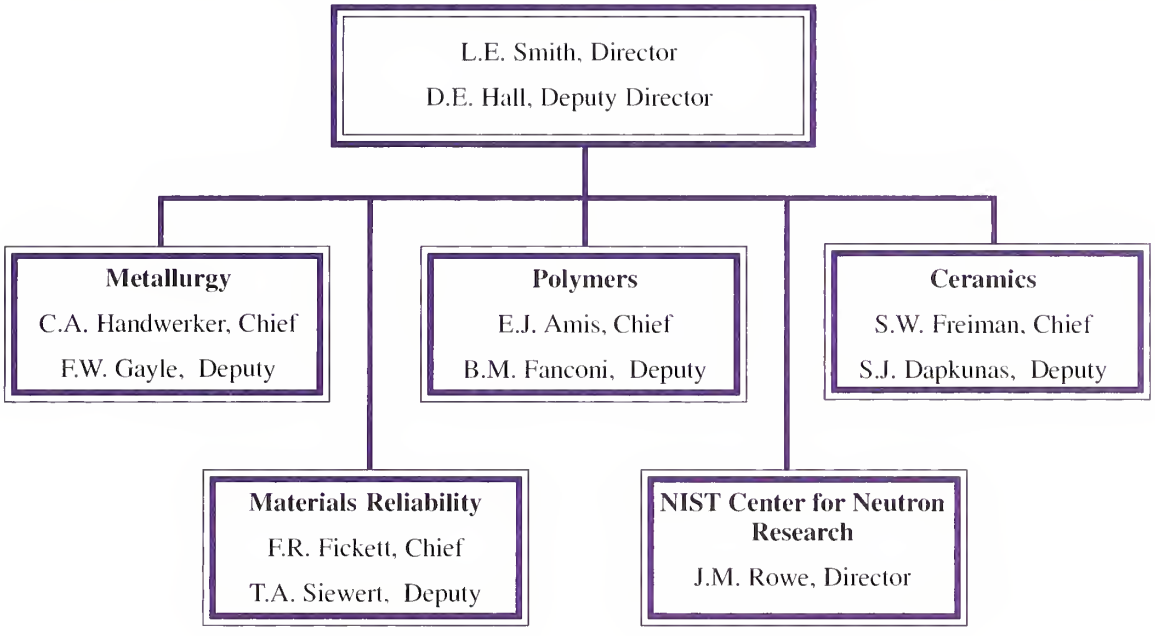


Materials Science and Engineering Laboratory



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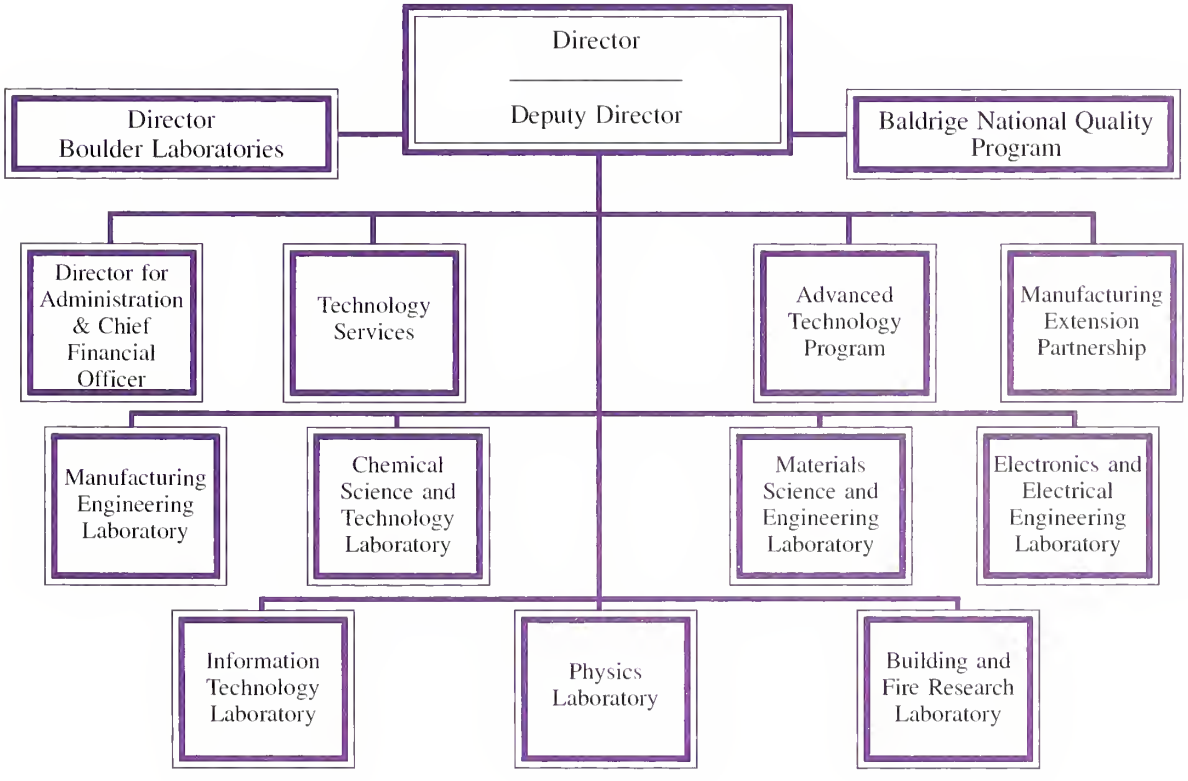

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